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# ABSTRACTS OF CURRENT PUBLISHED INFORMATION ON NICKEL AND ITS ALLOYS

## NICKEL

### Magneto-Optical Properties of Nickel

G. S. KRINCHICK and I. S. STROGANOVA: 'The Magneto-Optical Properties of Iron, Nickel and Cobalt in the Ultra-Violet Region.'

*Fiz. Metal. Metalloved.*, 1959, vol. 7, No. 3, pp. 460–1. Translated in *Physics of Metals and Metallography*, vol. 7, No. 3, pp. 139–40.

The magneto-optical data presented were derived from measurements, in the ultra-violet region of the spectrum, carried out to extend the range of frequencies in which information is available on the dynamic properties of ferromagnetics.

### Influence of Plastic Deformation on the Coercive Force of Nickel and Nickel-Iron Alloys

E. KNELLER and G. SCHMELZER: 'The Coercive Force of Nickel and Nickel-Iron Alloys During Plastic Deformation.'

*Zeitsch. f. Metallkunde*, 1960, vol. 51, June, pp. 342–9.

The coercive force of single- and polycrystals of nickel and nickel-iron alloys (containing 50–100 per cent. nickel) was measured as a function of plastic deformation.

In the case of single crystals, the coercive-force/yield-stress curve is characterized by three regions (varying in slope) which correspond to the three regions of the work-hardening curve. The increase in the coercive force during deformation is discussed in terms of the magnetoelastic interactions between magnetization and the dislocations induced during the plastic deformation.

### Influence of Hafnium Additions on the Properties of Nickel

R. REINBACH: 'Hafnium as an Alloying Element in Copper, Iron and Nickel.'

*Zeitsch. f. Metallkunde*, 1960, vol. 51, May, pp. 292–4.

The electrical resistance, thermal EMF, coefficient of thermal expansion, and the hardness of copper-hafnium alloys (containing 0·1–2 per cent. hafnium), iron-hafnium alloys (containing 0·1–2 per cent. hafnium) and nickel-hafnium alloys (containing 0·1–8 per cent. hafnium) were studied as a function of hafnium content.

The alloying effects of hafnium were found to be similar to those of titanium and zirconium. Worthy of note are considered the appreciably higher recrystallization temperature and resistance of the alloys *vis-à-vis* those of the basis metals. Limits of solubility of hafnium in copper, iron and nickel were, respectively, 0·2, 0·2 and 2 wt. per cent.

### Cementation of Copper with Nickel in the Ammoniacal Region

R. SCHRADER and D. CLAUSS: 'The Cementation of Copper with Metallic Nickel in the Ammoniacal Region.'

*Jnl. Praktische Chemie*, 1959, vol. 9, pp. 194–200.

The authors show, on the basis of thermodynamic calculations, that the cementation reaction of copper with nickel, which proceeds spontaneously in weakly-acidic solutions, is also possible, despite the presence of complexes, in the ammoniacal region.

Details are then given of two techniques for the production of nickel powders active in an ammoniacal medium (reduction of $NiCO_3$ with town's gas, and hydrogen reduction of ammoniacal nickel solutions under pressure in an autoclave): the capacity of such powders (i.e., the extent to which they are cemented) lies in the region of 94 per cent.

The paper ends with a description of apparatus which enables the conventional discontinuous cementation process to be replaced by a continuous flow-through process. Using this procedure, the copper concentration of an ammoniacal nickel liquor can be reduced to $10^{-5}M$ (0·7 mg./L.).

### Hydrogen/Deuterium Exchange on Nickel

G. M. SCHWAB and E. KILLMANN: 'The Mechanism of the Hydrogen/Deuterium Exchange on Nickel.'

*Zeitsch. Physikalische Chemie*, 1960, vol. 24, Apr., pp. 119–29.

The kinetics of the hydrogen/deuterium exchange on nickel film were studied in the present investigation at temperatures in the range 100°–165°C. The apparent activation energy was 7·5 kcal./mol. The rate of reaction, as a function of deviation from equilibrium conditions, was logarithmic. The initial rate was dependent (in an unsymmetrical manner) on the partial pressures of the hydrogen and deuterium. The results are considered best explicable in terms of a bimolecular interchange between adsorbed molecules (atom pairs), a conclusion which is in agreement with the authors' previous assumptions.

### Thermodynamic Properties of Dilute Solutions of Sulphur in Liquid Nickel, Nickel-Iron Alloys and Cobalt-Nickel Alloys

C. B. ALCOCK and L. L. CHENG: 'A Thermodynamic Study of Dilute Solutions of Sulphur in Liquid Iron, Cobalt and Nickel, and Binary Alloys between these Metals.'

*Jnl. Iron and Steel Inst.*, 1960, vol. 195, June, pp. 169–73.

The authors report a study of the thermodynamic properties (at 1540°C.) of dilute solutions of sulphur

in liquid iron, cobalt and nickel, and in liquid iron-cobalt, cobalt-nickel and nickel-iron alloys.

In the experimental technique used (that described by ALCOCK and RICHARDSON in *Acta Metallurgica*, 1958, vol. 6, pp. 385–95) small beads of the metals and alloys were brought to equilibrium with $H_2/H_2S$ mixtures of controlled ratio. The sulphur content of the beads was then analyzed by the oxygen-combustion method.

Alcock and Richardson showed that the partial free energy of solution of sulphur in dilute solution in liquid nickel was about 2·5 kcal. *more* negative than that in super-cooled iron at 1500°C., and about the same amount *less* negative in the case of liquid cobalt compared with iron. The present research has confirmed this sequence of stabilities for the dilute solutions of sulphur in the liquid iron-group metals, and has further demonstrated that, whereas sulphur departs measurably from Henry's law in dilute solution in liquid iron, the solutions in liquid nickel conform to this law up to at least 6 at. per cent. In the liquid-cobalt solvent the maximum sulphur content which was studied was about 1 at. per cent., and no deviation from Henry's law could be found.

The results for the variation of the activity coefficient of sulphur across the whole composition range of the binary alloys are discussed in terms of a simple theory, and are compared with those for other dilute solutes in these alloys.

### Dissolution of Nickel in Acidified Ferric and Ceric Solutions

P. M. CHRISTOPHER and C. V. KING: 'Dissolution of Nickel in Acid Ferric and Ceric Solutions.'

*Jnl. Electrochemical Soc.*, 1960, vol. 107, June, pp. 493–5.

The dissolution rates of nickel cylinders rotated in acidified solutions of $FeCl_3$ and $Ce(SO_4)_2$ (the ferric solutions were made up with 1*M* HCl, the ceric with 1*M* $H_2SO_4$) were determined at 18°–21°C.

The rates were found to be transport-controlled, but the polished nickel surface became very rough (especially in the ferric solution), and first-order constants were obtained only when a uniform degree of roughness was maintained during runs. The values of the rate constants were then dependent on the condition of the metal (e.g., resulting from annealing, mechanical working, etc.).

The roughening of the nickel is considered at least partly due to preferential dissolution at grain boundaries and at flaws in the mechanically-worked metal. This effect is considered to indicate that, while the cathodic reaction (reduction of ferric or ceric ions) probably occurs equally well at any point on the surface, the anodic reaction (formation of aqueous $Ni^{++}$) is not uniformly distributed. The rough surface of a rotating cylinder increases turbulence in the solution, at least near the interface, resulting in larger dissolution rates than are found with smooth cylinders.

### Cold Rolling of Solid Sections by the Pilger Process

R. HINKFOTH: 'Cold Rolling of Solid Sections by the Pilger Process.'

*Neue Hütte*, 1959, vol. 4, Jan., pp. 40–5.

The factors involved in the rolling of tubes by the Pilger process are outlined, the advantages and limitations of the process (*vis-à-vis* cold drawing) are discussed, and Pilger mills and their operating principles are described. In the remainder of the paper, the author reports the results of experiments carried out, using iron and aluminium specimens, to investigate the feasibility of applying the process to the production of rod. Modifications necessitated in the operation and construction of the mill are considered, and finally details are given of exploratory studies of the Pilger rolling of nickel rod.

*See also*

### Cold Rolling of Nickel Rod by the Pilger Process

R. HINKFOTH: 'Cold Rolling of Nickel Rod by the Pilger Process.'

*Neue Hütte*, 1959, vol. 4, May, pp. 420–4.

Subsequent to exploratory studies indicating the feasibility of applying the Pilger process to the cold rolling of nickel (*see previous abstract*), the author extended his experiments, with the aim of perfecting a technique capable of (1) achieving a 70 per cent. reduction in nickel rod 22 mm. in diameter, and (2) suitable for application on a commercial basis. The experiments, the data and operating conditions determined, and the difficulties encountered are outlined in the present paper.

In the final section of the paper the author discusses the possibility of applying the Pilger process to the rolling of rod less than 22 mm. in diameter.

### Determination of Nickel in Soil and Laterites

A. N. CHOWDHURY and B. DAS SARMA: 'Improved Rapid Determination of Nickel in Soils and Laterites.'

*Analytical Chemistry*, 1960, vol. 32, June, pp. 820-1.

In the rapid geochemical method for the colorimetric estimation of nickel outlined by the authors, nickel in solution is separated from most other metals by extraction of nickel dimethylglyoxime, using a mixed solvent containing benzene and amyl alcohol. The separated organic layer is shaken with strong alkali and more dimethylglyoxime, which extracts nickel again in the aqueous layer and develops the wine-red colour. The colour is then compared with standards previously prepared.

Comparison of the results with those obtained by conventional methods shows that the error is less than 30 per cent. for nickel present in the range 50–4,000 p.p.m. In an 8-hour working day it is possible to carry out 30 to 40 estimations with an accuracy in the region of 20 per cent.

### Photometric Determination of Cobalt in Nickel

C. L. LUKE: 'Photometric Determination of Traces of Cobalt in High-Purity Nickel.'

*Analytical Chemistry*, 1960, vol. 32, June, pp. 836-7.

Although the direct photometric nitroso-R method for determination of trace amounts of cobalt in nickel has proved reliable, and is rendered attractive by its reasonably good selectivity, the technique is not very sensitive because of the limitations placed on the size of the sample. Separation of the cobalt from a large sample of the nickel before attempting the photometric determination appeared to offer a solution to the problem of increasing the sensitivity, and, on the basis of the author's recent work on the determination of chromium in nickel (*ibid.*, 1958, vol. 30, Mar., pp. 359–61: see abstract in *Nickel Bulletin*, 1958, vol. 31, No. 5, p. 127), it appeared probable that the separation could be accomplished by first converting the cobalt to a very stable cobaltic ammine, and then removing the nickel by precipitation as hexamminoperchlorate. This assumption has been confirmed, and the procedure evolved (outlined in this paper) is stated to have increased the sensitivity of the nitroso-R method markedly without any decrease in accuracy.

The proposed new method is considered suitable for the determination of 0·001–0·02 per cent. of cobalt in high-purity nickel, and, by increasing the sample size and the light path in the photometric measurement, it should be possible to determine as little as 0·0001 per cent. It is suggested that the method can probably be used for the determination of small amounts of cobalt in high-nickel steels, provided that the bulk of the iron is removed by an ether extraction before the precipitation of the nickel.

## ELECTRODEPOSITION AND OTHER COATING METHODS

### Influence of Urea on Electrodeposition of Nickel

W. MACHU and M. F. M. EL-GHANDOUR: 'Influence of Urea on the Electrodeposition of Nickel.'

*Werkstoffe u. Korrosion*, 1960, vol. 11, May, pp. 283–6.

Previous studies by the authors had demonstrated that urea and ammonium sulphate markedly influence the electrodeposition of chromium, iron and iron-chromium alloys. In the experiments now described they have extended the scope of their investigations to the determination of the effects of urea on the electrodeposition of nickel.

A solution of the following composition was selected as a basis for study: $NiSO_4.7H_2O$ 112·3, $H_3BO_3$ 24·73, urea 180·18, g./L. The effects of varying the pH, temperature, current density and, in a few instances, the nickel concentration were determined.

The presence of urea gave rise to high polarizations, and the cathode efficiency was reduced to less than 50 per cent. Use of a higher temperature and current density only partially counteracted this effect.

On the other hand, addition of urea permitted a plating solution of low pH value (2·0–3·0) to be operated at very high current densities (20 amp./dm.$^2$). Satisfactory nickel deposits (with improved brightness, which could be increased to mirror brightness by addition of brightening agents) were obtained even at relatively low nickel concentrations (e.g., 140 g./L. of nickel sulphate).

### Raising the Standards of Nickel/Chromium Plating

'Your Chromium Will Soon Last Longer.'

*Reprint* from *Engineering*, 1960, vol. 189, June 3, pp. 742–3.

Issued by MOND NICKEL COMPANY, LTD., 1960; 2 pp.

See abstract in *Nickel Bulletin*, 1960, vol. 33, No. 7, p. 164.

### Correlation of Atmospheric-Corrosion, Accelerated-Corrosion and Service Tests on Nickel/Chromium-plated Components

R. B. SALTONSTALL and D. R. MILLAGE: 'Correlation of Outdoor-Exposure, Service and Accelerated Tests on Chromium-Plated Bright Work.'

*Plating*, 1960, vol. 47, June, pp. 637–44.

The success of efforts to improve the protective value of nickel/chromium electrodeposited coatings has been assessed mainly on the basis of the results of such corrosion tests as the 'Corrodkote' and copper-accelerated acetic-acid/salt-spray tests, and in terms of the corrosion behaviour of the deposits during stationary exposure to atmospheric environments. In view of doubts as to the advisability of using such tests as criteria of service performance, a research programme was initiated in the winter months of 1958–59 to correlate the results of accelerated-corrosion tests and 'stationary' and 'mobile' atmospheric tests.

Test panels, plated as indicated below, were exposed (1) on a roof in a Detroit industrial atmosphere, (2) to the 'Corrodkote' and copper-accelerated acetic-acid/salt-spray tests, (3) on the front bumpers of cars driven in various cities. The data derived from these tests are correlated in the paper (which also includes photomicrographs illustrating the types of corrosion observed).

*Steel Panels*

(*a*) 1 mil of bright nickel + 0·01 mil of chromium*.

(*b*) 1 mil of bright nickel + 0·03 mil of HT-HR** chromium.

(*c*) 1 mil of nickel (80 per cent. sulphur-free semi-bright + 20 per cent. bright nickel) + 0·01 mil of chromium.

(*d*) 0·4 mil of copper + 0·8 mil of bright nickel + 0·03 mil of HT-HR chromium.

*Zinc-Die-Cast Panels*

(*e*) 0·4 mil of copper + 0·8 mil of bright nickel + 0·01 mil of chromium.

(*f*) 0·4 mil of copper + 0·8 mil of bright nickel + 0·03 mil of HT-HR chromium.

* Conventional chromium, 110°F., ratio 100.

** High-temperature (130°F.), high-ratio (135-165).

(*g*) 0·4 mil of copper + 0·03 mil of HT-HR chromium + 0·8 mil of bright nickel + 0·03 mil of HT-HR chromium.

(*h*) 0·4 mil of copper + 0·8 mil of nickel (80 per cent. sulphur-free semi-bright + 20 per cent. bright nickel) + 0·01 mil of chromium.

The results of these exploratory tests are considered to justify the following conclusions:

'1. The CASS and 'Corrodkote', as well as static roof exposure, were all appreciably more severe on the 1·0 mil bright nickel + 0·1 mil chromium than one winter on the mobile test sites.

'2. Neither the CASS, 'Corrodkote', nor static roof exposure correctly predicted that the effect of 0·03 mil HT-HR chromium, compared with 0·01 mil conventional chromium on 1·0 mil bright nickel, at the end of one-year service exposure would be harmful.

'3. The marked superiority of the composite nickel coatings (80 per cent. sulfur-free+20 per cent. bright nickel), compared with an equal thickness of bright nickel, which was apparent in the accelerated tests, was less apparent in our roof exposure. The mobile-exposure results for both of these systems, however, showed very little deterioration in one winter exposure, and longer mobile tests are needed to determine comparative performance.

'The unexpectedly good behaviour of the bright nickel with 0·01 mil chromium plate on the mobile test sites tends to confirm an observation made previously, viz., that failure to specify adequate bright-nickel thickness, and/or failure to enforce specifications, has resulted in untimely failures.

'The danger in translating or interpolating correlation between accelerated tests and service exposure from one coating system to another, or even within the same system when variables in types of coating are introduced, has been pointed out many times. In spite of this, there is an understandable but highly undesirable tendency to specify the new accelerated tests for many coatings and materials without proper correlation. This practice should be discouraged to the greatest extent possible.'

### Experience in Copper/Nickel/Chromium Plating of Zinc-base Die Castings

J. CHADWICK: 'Experiences in the Decorative Plating of Zinc-Alloy Die-Cast Components.'

*Electroplating and Metal Finishing*, 1960, vol. 13, July, pp. 239–45.

This review, based on the author's experience in copper/nickel/chromium plating zinc-base die castings in automatic plants, covers the causes of, and the procedures found suitable for eliminating, difficulties involved in (1) cleaning the basis metal prior to plating, (2) copper plating, (3) bright-nickel plating, and (4) chromium plating.

The bright-nickel-plating stage of the sequence forms the main subject of the review. It is emphasized that, provided the cleaning and copper-plating stages are satisfactorily carried out, little difficulty should be encountered in nickel plating. Defects do, however, arise, and the author discusses his experience in the prevention or elimination of the following: roughness in the deposit; pitting; mistiness; unsatisfactory adhesion; metallic and organic contamination of the plating solution; blistering; variation in the thickness of the deposit.

In the final section of the paper the author draws attention to the improvements in corrosion-resistance inherent in the advent of methods of producing duplex-nickel coatings and thicker chromium overcoatings. His recommendations for a plated finish 'to withstand several years' outdoor service and yet be suitable for mass-production purposes' are:

| | | |
|---|---|---|
| Copper .. .. | 0·0003 in. | 0·0075 mm. |
| Semi-bright levelling nickel .. .. | 0·0008 in. | 0·02 mm. |
| Bright nickel .. | 0·0002–0·00031 in. | 0·005–0·00775 mm. |
| Chromium (deposited by an approved high-temperature method) .. .. | 0·00002 in. | 0·0005 mm. |
| | (0·00003 in. for relatively simple shapes) | 0·00075 mm. |

### Electroless Plating of Large-Scale Components by the 'Kanigen' Process

W. J. CREHAN: 'Chemical Nickel Plating on a Large Scale by the 'Kanigen' Process.'

*Amer. Soc. Mechanical Engineers*, 1959, *Paper* 59-*A*-286; 8 pp.

In the four years during which the Sharon chemical-nickel-plating plant of the General American Transportation Corporation has been in production, many different kinds of equipment (mainly of a type which could not have been electroplated) have been plated with nickel-phosphorus alloy by the 'Kanigen' process. The facilities provided are capable of handling larger components than any other plant in the world, and the plating of the interior surfaces of railroad tank cars offers, for example, relatively little difficulty. In this paper the author reviews the procedures developed for nickel plating on such a large scale, discusses the problems involved, considers the properties of the chemically-deposited nickel-phosphorus coating, and draws attention to the fields in which 'Kanigen' plating has found application.

The review opens with notes on the surface preparation of large welded vessels and large castings, on plating techniques for coating the interior and exterior surfaces of large components, and on fixturing. The economic advantages of 'Kanigen' plating are then touched on, and consideration is given to the properties of the coating, and the effects of 'Kanigen' plating on the fatigue life of the basis metal. Finally the applications of the chemical-plating process are reviewed in terms of its use:

(*a*) to confer protection against corrosion or product contamination;

(*b*) for hard coating;

(*c*) to prepare aluminium or stainless steel for soldering or brazing;

(*d*) to prevent galling in metals;

(*e*) to prevent stress corrosion in stainless steel.

### Automatic Control of the pH of Nickel-Plating Solutions

G. GABRIELSON: 'Automatic pH Regulation of Nickel-Plating Solutions.'

*Metal Finishing Jnl.*, 1960, vol. 6, June, pp. 239–42.

The effects of variations in the pH value of nickel-plating solutions are discussed, the factors involved in the automatic regulation of the pH to a desired value are considered, and finally details are given of the principles underlying, and the equipment required for, the method of automatic regulation based on electrometric measurement of the pH of the solution.

### Electrodeposition and Properties of Nickel-Iron Films

See abstract on p. 202.

### Vapour Deposition of Molybdenum Coatings on Nickel or Nickel-Plated Basis Metal

S. T. WLODEK and J. WULFF: 'The Adhesion of Vapor-Deposited Molybdenum Coatings.'

*Jnl. Electrochemical Soc.*, 1960, vol. 107, June, pp. 565–8.

Following the development of a technique capable of plating large articles with molybdenum (involving vapour deposition by reduction of molybdenum pentachloride with hydrogen), a study was made of the factors affecting the adhesion of the molybdenum coating (e.g., accidental contamination of the pentachloride with oxychlorides, and the purity, composition and preparation of the basis metal). In the investigation described, the authors confined themselves to evaluation of the influence of the basis metal in relation to the adherence of molybdenum coatings deposited on (1) iron, carbon steels, 18–8 chromium-nickel stainless steel, copper, nickel, and 'Inconel', and (2) iron (and, in some cases, low-carbon steel) electroplated with nickel, cobalt, copper, chromium or rhodium.

Molybdenum deposits exhibiting satisfactory adhesion could not be obtained on pure iron, stainless steel, low-carbon steel or chromium-plated surfaces, but coatings deposited on nickel, copper, 'Inconel', or any of the nickel-, cobalt-, copper-, or rhodium-plated iron or steel samples exhibited excellent as-plated adherence. These findings are discussed.

It is concluded that adherent coatings of molybdenum can be vapour-plated onto surfaces which are free from carbon, or from constituents that form more stable chlorides than molybdenum. The coating system must be such as to eliminate oxygen contamination. Prior electrodeposition of copper, cobalt, nickel or rhodium on basis metals containing carbon, iron or chromium ensures deposition of adherent coatings (provided that the basis metal is stable at the plating temperature). It is suggested that similar criteria may govern the adherence of chromium, tantalum or tungsten coatings produced by chloride reduction.

## NON-FERROUS ALLOYS

### Aluminium-Nickel-Silicon Brass Strips, Rods and Wire: Specification

MINISTRY OF AVIATION: 'Aluminium-Nickel-Silicon Brass Strips, Rods and Wire (Suitable for Springs).'

*Aircraft Material Specification D.T.D.* 5009, issued Feb. 1960. Price 1/-.

The specification is in three sections. Section I stipulates general requirements. Sections II and III (which are specific, respectively, to strip and to rods and wire) cover the conditions and dimensional tolerances of the material, and selection, preparation, testing and mechanical properties of test samples.

The following compositional limits are specified: copper 81–86, aluminium 0·7–1·2, nickel 0·8–1·4, silicon 0·8–1·3, iron 0·25 max., lead 0·05 max., tin 0·1 max., manganese 0·1 max., per cent., remainder zinc.

### Lattice Constants of Nickel-Titanium Alloys

H. P. STÜWE and Y. SHIMOMURA: 'Lattice Constants of the Room-Centred-Cubic Phases FeTi, CoTi, NiTi.'

*Zeitsch. f. Metallkunde*, 1960, vol. 51, Mar., pp. 180–1.

The lattice constants of alloys of titanium with iron, cobalt and nickel were determined, as a function of composition in the region of the phases FeTi, CoTi and NiTi, on specimens quenched from 1000°C. On the basis of the data obtained, all the atoms are deemed equally responsible for the lattice contraction reported for these phases.

### Distortions and Binding Forces in Nickel-base Solid Solutions

N. I. NOSKOVA and V. A. PAVLOV: 'An X-Ray Study of the Distortions and Binding Forces in the Crystalline Lattice of Nickel-based Solid Solutions.'

*Fiz. Metal. Metalloved.*, 1959, vol. 7, No. 3, pp. 400–4. Translated in *Physics of Metals and Metallography*, vol. 7, No. 3, pp. 75–9.

On the assumption that the establishment of a direct relationship between fine structure and the effects of alloying additions, heat-treatment and deformation would contribute to an understanding of hardening processes in solid-solution alloys, the authors investigated, as a function of heat-treatment and plastic deformation, the static and dynamic distortions of the crystal lattice caused by alloying nickel with 10–60 per cent. copper or 0·023–7 per cent. aluminium. Grain-size and second-order distortion were established in deformed alloys.

On the basis of the data tabulated, it is concluded that the static distortion which occurs on alloying nickel with copper or aluminium increases as the alloying addition is increased. The characteristic temperature increased with increase in aluminium and decreased with increase in copper. Plastic deformation (the alloys were filed to powder at room temperature) lowered the characteristic temperature of the 2·93 per cent. aluminium alloy and

raised that of the alloy containing 40 per cent. copper. In reviewing the behaviour of alloys under load, it is considered essential that account should be taken of the possibility of an increase or reduction in the characteristic temperature.

### Thermodynamic Properties of the Gold-Nickel Solid Solutions

M. J. ROWNEY: 'Properties of the Gold-Nickel Solid Solutions.'

*Jnl. Birmingham Metallurgical Soc.*, 1960, vol. 40, June, pp. 24–30.

In the application of the science of thermodynamics to the study of the processes of solution, diffusion and precipitation in metals and alloys, it is desirable to relate measured thermodynamic quantities, such as heat of solution, specific heat, entropy and free energy, to the actual behaviour of the constituent atoms, as far as this is possible. In the past the approach with regard to binary alloys has been by use of the quasi-chemical theory, in which the chemical bond energies between like and unlike pairs of nearest-neighbour atoms are considered, and the local atomic configuration is predicted according to whether the heat of solution is positive or negative. The earlier work investigating the validity of this approach was carried out with systems having negative heats of solution, indicating a tendency for superlattice or compound formation. Extensive investigations of the gold-nickel system, which has a positive heat of solution, have cast doubts on the generality of the quasi-chemical theory, and have demonstrated the necessity of taking into account differences in the sizes of the component atoms, even when the system exhibits extensive solid solution.

In the light of these introductory comments, the author reviews data available in the literature on the thermodynamic properties of the gold-nickel solid solutions, and discusses agreement or discrepancy between experiment and theory.

### Thermodynamic Properties of Dilute Solutions of Sulphur in Liquid Cobalt-Nickel Alloys

See abstract on p. 189.

### Volume Magnetostriction of Cobalt-Nickel Alloys

T. KANEKO: 'Volume Magnetostriction of Cobalt-Nickel Alloys.'

*Jnl. Physical Soc. Japan*, 1960, vol. 15, Mar., pp. 463–5.

Forced volume magnetostriction of cobalt-nickel alloys (containing 10, 20 or 30 at. per cent. of nickel) was measured, using resistance strain-gauges, at magnetic-field strengths up to 15,000 Oe.

The data show that the fractional volume change per Oersted, after technical saturation, was smaller than $1 \times 10^{-10}$/Oe. The results are considered consistent, at least qualitatively, with the change in Curie temperature with hydrostatic pressure determined by PATRICK (*Physical Rev.*, 1954, vol. 93, p. 384).

### Magnetic Properties of Nickel-containing Copper Alloys

F. G. PARKER, R. M. WINTER and J. A. LAPORTA: 'Magnetic Properties of Copper Alloys.'

*Proc. Amer. Soc. Testing Materials*, 1959, vol. 59, pp. 230–61.

Although copper alloys are generally considered to be non-magnetic, under certain conditions they exhibit definite magnetic properties (in general, associated with their iron content and the mode in which the iron is present), a characteristic which is undesirable in alloys intended for use in such equipment as compasses or magnetic devices. The magnetic requirements laid down differ with the given application, but various specifications limit the maximum magnetic permeability to values ranging from 1·02 to 2 cgs. units. The first part of the work described was undertaken to determine which of the commercially available alloys meet these specifications.

The mass susceptibility and permeability of specimens of the following types of alloy (in at least two conditions of temper) were determined: copper, brass, lead-bearing brass, tin bronze, phosphor bronze, 12 and 18 per cent. nickel silvers, 10 and 30 per cent. cupro-nickels, low- and high-silicon bronze, tellurium copper, 'Telnic' bronze, and 'Phosnic' bronze. The following generalizations are drawn from the data tabulated: (1) Stress-relief annealing does not result in any significant change in the susceptibility of cold-worked specimens; (2) For a given iron content, an increase in silicon content causes an increase in susceptibility; (3) Increase in nickel content tends to nullify the effects of iron (as, for example, reflected in the relatively low susceptibility of the 30 per cent. cupro-nickel, which contained approximately 0·6 per cent. of iron); (4) In $\alpha+\beta$ alloys susceptibility increases with increase in iron content. Other experiments were carried out on aluminium bronzes and copper alloys prepared with iron contents at or near the maximum specified.

In the second stage of the programme a systematic study was made of the effects of iron additions on the magnetic properties of copper, $\alpha$ brass and $\beta$ brass.

### Mechanism of Protective-Scale Formation on Nickel-containing Copper-base Alloys

J. C. BLADE and A. PREECE: 'The Mechanism of Protective-Scale Formation on Copper-base Alloys.'

*Jnl. Inst. Metals*, 1960, vol. 88, June, pp. 427–32.

In many cases the enhanced oxidation-resistance conferred on metals by certain alloying additions is associated with the formation of an oxide film of one of the alloying elements at the scale/metal interface. The available information indicates that such protective films are formed only when the alloying addition exceeds a certain critical value. The work reported by the authors was conducted to obtain more information on the factors governing this value.

The selection of alloys for investigation was influenced by the possible use of copper-base alloys in gas-turbine heat exchangers. Scale formation was therefore studied in binary or ternary alloys containing the following elements in the range stated:

| | % |
|---|---|
| Ni | 10·8-42·6 |
| Mn | 7·85-31·2 |
| Zn | 4·85-20·6 |
| Sn | 5·3-19 |
| Al | 0·5-5·5 |

2·5% Al + 8, 30 or 64% Ni
30% Ni + 0·5-3% Al

In the case of the Cu-2·5Al, Cu-5Ni-2·5Al, and Cu-30Ni-2·3 Al alloys, a study was made of the effects, on scaling properties, of minor additions of the following elements: silicon, manganese, beryllium, chromium, titanium, thorium or cerium. Specimens were oxidized by exposure (for 48-1000 hours at temperatures in the range 500°-1000°C.) to an atmosphere obtained by burning paraffin with fifty times its own weight of air. The specimens were exposed in the as-cast, hot-rolled, hot-rolled-and-solution-treated, or solution-treated-and-cold-rolled conditions.

From the data and photomicrographs presented, the authors draw conclusions which they summarize as follows:

'The chief criterion that determines the minimum quantity of alloying element necessary for the formation of a protective oxide layer at the scale/metal interface appears to be the rate at which the solute atoms can diffuse to the interface and be preferentially oxidized there. For the binary alloys examined, a linear relationship of the form $C \propto 1/DV$ determines the approximate value of the critical concentration $C$ of the solute, where $D$ is its diffusion coefficient and $V$ its valency.

'In ternary alloys, not only does the total quantity of solute atoms capable of preferential oxidation have to be taken into account, but also the possibility that diffusion coefficients of the various solutes may be very different in the ternary alloys from those found in the respective binary alloys. Little is known of diffusion coefficients in ternary copper-base alloys. The results now obtained for the copper-nickel-aluminium alloys do, however, give some indication of the effect of the interplay of these factors for this system, e.g., in 48-hour scaling tests, the addition of 30 per cent. nickel to the binary copper-2·5 per cent. aluminium alloy reduces the temperature where heavy scale formation begins from 1000° to 800°C. This may indicate that the diffusion coefficient of aluminium in the ternary alloy is less than in the simple binary copper-aluminium alloy.

'Minor additions to copper-aluminium and copper-nickel-aluminium alloys, such as 1 per cent. chromium, have a beneficial effect on the resistance to scaling. The addition of 0·5 per cent. chromium is sufficient to produce a high resistance to oxidation, except for the alloy containing 30 per cent. nickel, where 1 per cent. chromium is necessary. In alloys containing up to 5 per cent. nickel, the addition of 2 or 4 per cent. silicon or 0·5 per cent. beryllium appears useful in increasing the resistance to scaling.

'The mechanism by which additions influence oxidation behaviour is not clear, since insufficient diffusion data, particularly with reference to the effects of small amounts of a second solute, are available.

'Precipitation, by heat-treatment, of an alloying element that confers resistance to oxidation leads to increased oxidation rates in the heterogeneous alloy.'

### Creep and Rupture Properties of 'Ni-Vee' Bronze

H. V. KINSEY: 'The Creep and Rupture Properties of Three Cast Copper-base Alloys Between 550° and 650°F. (290° and 345°C.).'

*Proc. Amer. Soc. Testing Materials*, 1959, vol. 59, pp. 940-51.

The investigation described was initiated (at the Mines Branch, Physical Metallurgy Division, Canadian Department of Mines and Technical Surveys) as part of a programme to study the foundry characteristics and the properties of various types of bronze. In the phase of the programme reported, the creep and rupture properties of three grades of bronze were investigated (see composition below).

'Ni-Vee C' and 'Navy M' were tested in the as-cast condition; 'Ni-Vee A' was evaluated both in the as-cast condition and after solution-treatment for 5 hours at 1400°F. (760°C.) followed by oil quenching and ageing for one week at 550°F. (290°C.). Creep and stress-rupture tests were conducted at 550°, 600°, 650° and 700°F. (290°, 315°, 345° and 370°C.). Tensile properties were determined at room temperature. Also reported are data available on the tensile properties of some of the melts at 550°, 600° and 650°F.

| Alloy | Cu % | Sn % | Pb % | Zn % | Ni % | Fe % | P % |
|---|---|---|---|---|---|---|---|
| 'Ni-Vee A' .. .. .. | 85·0-90·0 | 4·5-6·0 | 0·10 max. | 2·5 max. | 4·5-6·0 | 0·25 max. | — |
| 'Ni-Vee C'* .. .. .. | 80 | 5 | 5 | 5 | 5 | — | — |
| 'Navy M' .. .. .. | 86·0-90·0 | 5·5-6·5 | 1·0-2·0 | 3·0-5·0 | 1·0 max. | 0·25 max. | 0·05 max. |

* Nominal composition.

In discussing the extensive data presented, the author points out that inconsistencies in the test bars employed permitted no more than an indication of probable trends in the properties of the alloys. It is suggested, however, that creep and rupture properties are very sensitive to melting and casting conditions. The following general conclusions are drawn:

(1) In order of decreasing stress-rupture strength over the temperature range studied, the alloys tested are listed as follows:

'Ni-Vee A', as-cast;
'Ni-Vee C', as-cast;
'Ni-Vee A', solution-treated-and-aged;
'Navy M'.

(2) Solution and ageing treatment of the as-cast 'Ni-Vee' alloy did not affect its creep properties, but reduced its rupture strength.

(3) In order of decreasing creep-resistance at the test temperatures, the alloys are listed as follows:

'Ni-Vee A' (both conditions);
'Ni-Vee C';
'Navy M'.

**High-Temperature Properties of 'Cufenloy 30' in the Drawn-and-Stress-Relieved Condition**

W. F. SIMMONS, B. J. SIROIS, D. N. WILLIAMS and R. I. JAFFEE: 'Properties of 70-30 Copper-Nickel Alloy at Temperatures Ranging up to 1050°F.'

*Proc. Amer. Soc. Testing Materials*, 1959, vol. 59, pp. 1035–45; disc., pp. 1046–51.

Copper-nickel-alloy tubes have been used successfully for many years in heat exchangers and unfired pressure vessels operating at elevated temperatures and pressures. Design factors based on the properties of annealed material have, however, limited the 70-30 copper-nickel-alloy tubes to applications and designs which have failed to take full advantage of the higher mechanical properties attainable in the alloy. The investigation outlined in this paper was initiated to determine the feasibility of developing mechanical properties capable of meeting more stringent requirements in heat-exchanger applications. The particular objective was to produce tubes in the 70-30 alloy which, while exhibiting improved properties, would retain sufficient ductility to permit U-bending over small radii and expansion into tube sheets.

'Cufenloy 30', the alloy evaluated, contained copper 70, nickel 29·1, iron 0·5, manganese 0·35, lead 0·01, per cent. A considerable quantity of tubes were fabricated in this alloy, process-annealed to the average grain-size prescribed in *A.S.T.M. Specification B* 111, and subsequently drawn (with various degrees of cold reduction) to tubes ranging in diameter from $\frac{5}{8}$ to 1 in. (15 to 25 mm.) and with wall thicknesses up to 0·12 in. (3 mm.). The tubes were then heat-treated to a ductility which permitted them to be expanded into tube sheets and bent over small radii. Tubes exhibiting the following minimum mechanical properties at room temperature were sufficiently ductile to satisfy these requirements:

| | *p.s.i.* | *t.s.i.* | *kg./mm.*$^2$ |
|---|---|---|---|
| Tensile strength .. | 72,000 | 32 | 50·5 |
| Yield strength .. | 50,000 | 22·5 | 35 |
| Elongation in 2 in. (5 cm.): | | | |
| For wall thicknesses up to 0·049 in. (1·24 mm.) inclusive .. .. .. | | | 12 per cent. |
| For wall thicknesses over 0·049 in. .. .. .. | | | 15 per cent. |
| Expansion of tube inside diameter with tapered pin .. | | | 20 per cent. |

In addition to satisfying the standard mercurous-nitrate test, specimens of tubes U-bent over a radius equal to twice the diameter of the tube were subjected to a moist-ammonia atmosphere for ten months, under various conditions of stress, with no signs of stress-corrosion cracking.

The data presented by the authors in this paper were derived from a series of tests carried out to determine (on specimens from rod stock treated to develop the minimum properties listed above) the following properties of the alloy in the drawn-and-stress-relieved condition: thermal expansion; thermal conductivity; electrical resistivity; dynamic modulus of elasticity; tensile properties in the range 75°–1050°F. (24°–565°C.); and creep and stress-rupture properties in the range 500°–1050°F. (260°–565°C.).

On the basis of these data, the authors conclude that the combination of strength, ductility and stress-corrosion-resistance exhibited by the alloy renders it suitable for use in unfired-pressure-vessel applications at temperatures up to the region of 850°–900°F. (455°–480°C.).

**Properties of Cast 70-30 Cupro-Nickel**

B. F. SHEPHERD: 'Cast 70/30 Cupro-Nickel: Inherent Characteristics.'

*Modern Castings*, 1960, vol. 37, May, pp. 120–30.

The need for a material of high strength/weight ratio, capable of being used for thick-wall (⩾2-in.) pump castings, and of withstanding the higher pump pressures involved in severer service conditions, has focused attention on the properties obtainable in 70–30 cupro-nickel castings. The comprehensive investigation of the properties of 70–30 cupro-nickel described in this paper was undertaken to determine the feasibility (using *MIL-C*-20159 as a basic specification) of employing the alloy in high-pressure pumps.

The influence of the following factors was studied (and the results are reported in the paper): silicon content; niobium+silicon contents; cooling rate in the mould; homogenization heat-treatment; the mass of the test bar. The mechanical properties of test pieces taken from various locations in 70–30 cupro-nickel pump castings were determined, and welding characteristics and foundry control were investigated. Details are given of test procedures, and the data obtained are fully recorded.

Properties were found to be markedly affected by silicon content: the level of silicon required to meet the tensile properties laid down by MIL-C-20159 adversely affected weldability, while satisfactory weldability was contingent on low-silicon contents which were not compatible with the required tensile properties.

Addition of both niobium and silicon resulted, however, in the production of a weldable alloy with mechanical properties much higher than could be obtained with silicon alone. The silicon and niobium contents required varied with the strength/ductility level desired: a yield strength of 40,000 p.s.i. (18 t.s.i.: 28 kg./mm.$^2$) was achieved by silicon and niobium contents of about 0·45 per cent. each. Weldability at higher silicon contents was attained with a silicon content less than the niobium; higher strength at lower silicon levels was associated with a niobium content greater than the silicon.

Mechanical properties were markedly influenced by cooling rate in the mould, an effect which was reflected in the data derived from tests on actual castings and test bars of varying mass. While the 70-30 cast cupro-nickel alloy is not considered heat-treatable, it was found that mechanical properties can be substantially increased by homogenizing heat-treatment (the effects, in this connexion, of time, temperature and composition are discussed). The inherent potentiality of a given composition is realized by homogenization treatment, and results show that this heat-treatment can be used to neutralize the effects of differences in cooling rate.

Irrespective of composition and/or acceptable heat-treatments, ductility and Brinell hardness are shown to be directly related to the yield strength.

Production heats of the niobium-containing alloy have been made in induction and direct-arc furnaces. Direct-arc heats of 6,300 lb. (285 kg.) have been melted, utilizing up to 100 per cent. returns.

### Mechanical and Physical Properties of 20 per cent. Nickel Silver

W. H. JOHNSON and J. G. KURA: 'Mechanical and Physical Properties of Five Copper-base Casting Alloys.'

*Amer. Soc. Testing Materials*, 1960, *Preprint* 77; 15 pp.

The paper contains the data derived from measurement of the mechanical and physical properties of sound test bars from good-quality melts of the following commercial alloys (identified as 5B, 8A, 8C, 11A and 13B in *A.S.T.M. Specification B*30-59): 76Cu-2½Sn-6½Pb-15Zn alloy; 20 per cent. nickel silver; 81-4-15 silicon-brass; manganese bronze (65,000 p.s.i.); manganese-bronze (110,000 p.s.i.).

The properties determined ( at various temperatures in the range −40° to +550°F. (−40° to +290°C.)) were: ultimate strength, yield strength, elongation, reduction-in-area, modulus of elasticity, compressive strength, Charpy V-notch impact strength, Brinell hardness, fatigue resistance, machinability, melting range, patternmaker's shrinkage, density and specific gravity, electrical resistivity, thermal conductivity, and thermal expansion.

## NICKEL-IRON ALLOYS

### Magnetism and Magnetic Materials: Fifth International Conference, 1959

The *Proceedings of The Fifth Symposium on Magnetism and Magnetic Materials* held, under the aegis of The American Institute of Electrical Engineers, in Detroit, November 1959, are published in a *Supplement* (420 pp.) to the *Journal of Applied Physics*, 1960, vol. 31, May. Some idea of the range of subjects covered may be gained from the section heads under which the 170 papers are classified:


| | |
|---|---|
| Magnetism, General and Theory | pp. 3S–27S |
| Garnets | pp. 30S–55S |
| Permanent Magnets | pp. 58S–84S |
| Spin Waves and Magnetostatic Modes | pp. 86S–108S |
| Computers and Switching | pp. 110S–35S |
| Anisotropy | pp. 137S–60S |
| Techniques and Devices | pp. 162S–96S |
| Resonance | pp. 198S–226S |
| Soft Magnetic Materials | pp. 228S–49S |
| Ferrites and Oxides | pp. 251S–78S |
| Magnetic Films | pp. 279S–308S |
| Metals and Alloys | pp. 310S–31S |
| Magnetic Salts | pp. 336S–48S |
| Magnetic Compounds and Neutron Diffractions | pp. 350S–74S |
| Ferrimagnetic Resonance Effects and Miscellaneous | pp. 376S–414S |


The scope of papers relevant to nickel-containing materials is indicated in the abstracts below.

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S. SHTRIKAMN and D. TREVES: 'On the Remanence of Ferromagnetic Powders', pp. 58S–66S.

In this study of the maximum remanence of assemblies of aligned particles, maximum remanence in any direction is discussed and a relationship between its parallel and transverse components derived. Consideration is given to the special case of a rotationally symmetric assembly of uniaxial particles, and formulae are derived which enable calculation of the distribution of the easy axes of the particles from the angular dependence of the maximum remanence. Measurements of maximum remanence are discussed. The data presented were obtained from experiments on 'Alnico', 'Ferroxdure' powder and 'E.S.D.' iron.

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T. O. PAINE and F. E. LUBORSKY: ' 'Interaction-Anisntropy' Model of the Structure of 'Alnico' Magnet Alloys', pp. 78S–80S.

The permanent-magnet properties of 'Alnico' alloys have been attributed to the shape anisotropy of an elongated single-domain magnetic precipitate oriented by cooling in a field. This paper reports measurements of the angular variation of the magnetic properties of highly directional crystal-oriented 'Alnico 5 D.G.', 'Columax' and 'Ticonal XX', which are inconsistent with the simple shape-anisotropy model. A domain 'interaction-anisotropy' model

is proposed which treats the structure as a complex interconnected single-domain network.

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K. J. KRONENBERG: 'Transmission Electron Diffraction of 'Alnico V', pp. 80S–2S.

Although electron metallography has been successfully applied to study of the complex structure of 'Alnico V' and related materials, their brittleness was thought to restrict the technique to surface investigations. In this paper the author demonstrates the feasibility of obtaining (by grinding, polishing and etching) 'Alnico V' specimens sufficiently thin for subjection to transmission electron metallography. Electron micrographs illustrate the efficacy of the procedure described.

The results confirm the two-phase structure determined earlier with replica techniques, and transmission diffraction revealed the crystal structures of both phases. Phase transformation was studied in samples at various stages of heat-treatment.

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K. J. KRONENBERG and M. A. BOHLMANN: 'Long-Term Stability of 'Alnico' and Barium-Ferrite Magnets', pp. 82S–4S.

A study was made of the variations in the remanence of the following permanent-magnet materials with time (up to 10,000 hours): the iron-nickel-aluminium alloy 'Alnico III', the iron-cobalt-nickel-aluminium alloys 'Alnico V' and 'Alnico VII', and the barium ferrites 'Indox I' and 'Indox V'.

The investigation has been reported in greater detail in *Wright Air Development Center, Tech. Report* 58-535: see abstract in *Nickel Bulletin*, 1960, vol. 33, No. 1-2, p. 10.

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C. L. KOLBE and D. L. MARTIN: 'Hot Working of 'Alnico V' Alloys', pp. 84S–5S.

The brittleness of 'Alnico' alloys, which renders them unworkable at room temperature and moderately high temperatures, is associated with a two-phase microstructure. At high temperatures these alloys usually transform into a ductile body-centred-cubic phase, a transformation which has been used as the basis for successful hot fabrication. The experiments described form an extension of previous work on the evaluation of the hot-working characteristics of vacuum- or air-melted alloys.

It is shown that careful processing enables certain of the alloys to be fabricated by extrusion, swaging, and rolling. After heat-treatment the permanent-magnet properties of the wrought grades of alloy were found to be comparable, and the mechanical properties superior, to those of cast specimens of the same alloy.

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E. M. GYORGY: 'Flux Reversal in Soft Ferromagnetics', pp. 110S–17S.

Aspects of flux reversal in soft ferromagnetic materials are interpreted in terms of domain-wall motion, non-uniform rotation and uniform rotation. Special emphasis is placed on polycrystalline, square-looped ferrites and thin 'Permalloy' films. The importance of geometric effects is illustrated in a review of detailed models for the uniform and non-uniform rotational processes. Specific limitations of the existing models are discussed, and possibilities for future advances are briefly outlined.

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A. V. POHM, A. A. READ, R. M. STEWART and R. F. SCHAUER: 'Operation of Magnetic-Film Parametrons in the 100- to 500-Mc Regions', pp. 119S–20S.

The aim of the paper is 'to show analytically that, with suitable 'Permalloy' films and proper fabrication techniques, high-performance, low-power-consumption, magnetic-film parametrons can be constructed for operation in the 100- to 500-Mc oscillating frequency range'. A method of fabricating magnetic-film parametrons with strip-line techniques is described. By using the two additional subharmonic phase states obtained by a bias reversal, capacitative coupling can be easily achieved without the necessity of connexions.

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T. R. LONG: 'Electrodeposited Memory Elements for a Non-Destructive Memory', pp. 123S–4S.

The procedure outlined by the author has been used to electrodeposit nickel-iron films onto a non-magnetic wire substrate to produce memory elements for a fast non-destructive memory.

Plating in the presence of a directed magnetic field establishes an anisotropy favouring circumferential orientation. Axial interrogation fields cause reversible rotations of less than 90°, and produce output signals across the ends of the wire. Alloy composition and plating conditions are shown strongly to affect the results: the amount of stress in the deposit, the residual stress in the substrate, pre-treatment of the substrate, the use of a suitable wetting agent, and agitation are vital factors in this respect.

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W. C. SEELBACH and J. R. KISEDA: 'Elastic-Switching Properties of Some Square-Loop Materials in Toroidal Structures', pp. 135S–6S.

Bobbin cores made from $\frac{1}{8}$-, $\frac{1}{4}$-, and $\frac{1}{2}$-mil '4-79 Mo-Permalloy' tape, prepared from the same heat of material, were driven in the set direction by current pulses with fast rise and fall times (~8 m$\mu$sec.) over a range of pulse widths from 40 m$\mu$sec. to 1 $\mu$sec. The magnitude and rate of change of induction relative to a reproducible reset state were observed, for elastic- and inelastic-switching modes, as a function of applied field.

The concept of an elastic-switching constant $S_{w(r)}$ is introduced and illustrated by the data presented. $S_{w(r)}$ values for 'Mo-Permalloy' ranged from 0·0374 Oe.-$\mu$sec. for $\frac{1}{8}$-mil tapes, to 0·0913 Oe.-$\mu$sec. for $\frac{1}{2}$-mil tape.

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E. R. CALLEN: 'Anisotropic Magnetization', pp. 149S-50S.

In the presence of magnetocrystalline anisotropy, the magnetization of a ferromagnet depends upon its orientation as well as upon the temperature. The author has established, both classically and quantum-mechanically, the temperature-dependences of the anisotropic parts of the magnetization for all symmetries and for ferro- and ferrimagnets in the internal field approximation.

The non-spherical terms are small at all temperatures in iron and nickel, but are appreciable in some uniaxial materials, and very large in many crystals of low Curie point and large anisotropy.

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K. P. BELOV, G. I. KATAYEV and R. Z. LEVITIN: 'Internal-Friction Anomalies in Ferromagnets and Antiferromagnets near the Curie Point', pp. 153S-6S.

The authors discuss internal-friction anomalies caused by the redistribution of spins within the domains under the action of elastic stresses.

Determination of the temperature-dependence of internal friction in ferromagnetic alloys ('Invar' alloys) and antiferromagnets (MnO, CoO and NiO) which exhibit a large spontaneous deformation of the crystalline lattice near the Curie point revealed sharp peaks in the internal-friction curves. This phenomenon is interpreted on the basis of thermodynamical theory of second-order phase transitions.

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J. C. SAGNIS, M. TEIG and R. L. WARD: 'Effect of Geometry on Thick-Film Toroids', pp. 190S-1S.

Previous investigations of magnetic circuits having demonstrated that thick film cores of certain sizes could not be completely switched if a concentrated drive winding was used, and that the induced voltages differed in waveshape as a function of the location of the sense windings with respect to the concentrated drive winding, the experiments now reported were initiated to study these effects in planar thick-film toroids photo-etched from $\frac{1}{4}$- and $\frac{1}{2}$-mil-thick 'Hy-Mu 80'. On the basis of the data obtained, the authors advance a qualitative explanation of the phenomena observed.

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R. L. WARD: 'Experimental Flux-Pattern Determination in Magnetic Cores', pp. 192S-3S.

Brief particulars are given of two techniques used to measure quiescent flux patterns in non-linear magnetic structures. The application of the techniques to measurement of these patterns in magnetic cores etched from '4-79 Mo-Permalloy' sheet (<1 mil thick) is described.

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R. F. SOOHOO: 'Initial-Susceptibility Spectra of 'Permalloy' Films', pp. 218S-9S.

The author reports the findings of a theoretical and experimental investigation of the initial susceptibility of 'Permalloy' films in the frequency range 100-10,000 Mc./sec. The exact boundary-value problem of a film placed in a rectangular cavity is solved, and the result is compared with the simpler solution obtained by means of the Bethe-Schwinger perturbation formula.

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I. P. KAMINOW: 'Volume Dependence of Ferrimagnetic Resonance Properties in $Ni_{1-x}Co_xFe_2O_4$', pp. 220S-1S.

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L. G. VAN UITERT, R. R. SODEN and F. W. SWANEKAMP: 'Resonance Line Widths of Sintered Nickel Ferrites Having Low Porosities', pp. 226S-7S.

Ferrimagnetic resonance-line-width ($\Delta H$) values are presented, and discussed, for a range of nickel ferrites produced by sintering.

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E. A. NESBITT, R. D. HEIDENREICH and A. J. WILLIAMS: 'A Necessary Factor for Heat-Treatment of the 'Permalloys' in a Magnetic Feld', pp. 228S-9S.

Single crystals of a 63-35-2 nickel-iron-molybdenum alloy were grown to a purity (0·0006 per cent. oxygen) sufficient to prevent response to heat-treatment in a magnetic field. In subsequent experiments the introduction of an oxidizing atmosphere during heat-treatment caused the same single crystals (which contained 0·0077 per cent. oxygen after oxidation) to respond to the magnetic-field heat-treatment. From these results, and confirmatory experiments on single crystals of 68-31·9-0·1 nickel-iron-magnesium alloys, it is concluded that oxygen is a necessary factor in the heat-treatment of the 'Permalloys' in a magnetic field, a finding which corroborates the results of work recently published on 'Perminvar' alloys.

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M. LAURIENTE and G. E. LYNN: 'Characteristics of 'Supermendur' at 500°C.', pp. 237S-8S.

The authors report, and discuss, an anomolous transient effect observed when the magnetic properties of 'Supermendur' toroidal tape cores were measured, in air at temperatures in the range 500°-600°C., using a constant current flux reset tester. This type of core tester normally establishes a steady-state cyclic-flux condition in a core within one cycle of excitation frequency, but it was found that the 'Supermendur' alloy required several hundred seconds to establish this condition.

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R. E. ALLEY and V. E. LEGG: 'Effects of Hydrostatic Pressure on the Properties of Magnetic Materials', pp. 239S-40S.

The data presented were derived from tests to determine the influence of hydrostatic pressure on the initial permeability and the hysteresis loop of such commercially important materials as: '4-79 Mo-Permalloy', 'Supermalloy', grain-oriented silicon steel, 'Supermendur', '2-81 Mo-Permalloy', carbonyl iron, and various ferrites (including Ni-Zn and Mn-Zn ferrites).

It is concluded that the properties of most solid materials are only slightly affected by pressures up to the maximum studied (20,000 p.s.i.: 9 t.s.i.: 14 kg./mm.$^2$). In the case of some types of Ni-Zn and Mn-Zn ferrite, the coercive force increased, and permeability decreased, with pressure.

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R. C. BARKER and R. M. BROWNELL: 'Direct-Current Measurements on Tape-Wound Cores', pp. 243S–4S.

The purpose of the paper is 'to indicate the sort of experimental results that can be expected of hysteresis-loop measurements under conditions of constant induced voltage, and to report some of the preliminary inferences that have been drawn'. The hysteresis-loop measurements reported were made on tape-wound cores of commercial-grade grain-oriented 50–50 nickel-iron material at very low domain-wall velocities.

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A. I. SCHINDLER and E. I. SALKOVITZ: 'Effect of Applying a Magnetic Field During Neutron Irradiation on the Magnetic Properties of Fe–Ni Alloys', pp. 245S–6S.

To investigate further the reported similarity between thermally-induced ordering and irradiation-induced ordering, four commercial alloys of nickel-iron base (including 'Permalloy' and 'Mumetal') were irradiated in the presence of a saturating magnetic field. The fact that square hysteresis loops were exhibited by all the samples irradiated is considered consistent with the proposal that neutron irradiation causes directional short-range ordering and consequent uniaxial anisotropy.

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A. C. MOORE and A. S. YOUNG: 'Some Physical Properties of Thin Magnetic Films', pp. 279S–80S.

The magnetic properties of thin (1000-Å) films of 80–20 nickel-iron alloy evaporated onto a glass substrate in a magnetic field can vary considerably. In the experiments reported a study was made of the properties of such films deposited onto a substrate at 300°C. (the temperature at which minimum coercivity in the preferred direction, $H_c$, occurs).

The data show that, under suitable evaporation conditions, the magnetocrystalline anisotropy is averaged out by the random array of small crystallites. The stress present in the films cannot be eliminated, but use of alloys whose mean magnetostriction is zero renders its effect insignificant. It is easier to obtain uniformity in the magnetic properties of a number of elements in a plane if the magnetic film is continuous and edge effects are excluded.

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W. DIETRICH and W. E. PROEBSTER: 'Millimicrosecond Magnetization Reversal in Thin Magnetic Films', pp. 281S–2S.

A description is given of a special pulse equipment (based on a pulse-sampling oscilloscope with an overall time resolution of 0·35 mμsec.) used to study the millimicrosecond flux reversal in thin 'Permalloy' films. Output signals as short as 1 mμsec. have been obtained and are discussed in relation to the underlying reversal processes.

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E. M. BRADLEY and M. PRUTTON: 'Magnetization Reversal in Uniaxial Films near to the Preferred Direction', pp. 285S–6S.

The authors report a study of the switching process occurring when fields are applied at an angle of 10° to the preferred direction of uniaxial thin magnetic films having almost ideal square hysteresis loops in the preferred direction, and almost straight-line loops in the 'hard' direction. (The experiments were carried out on films of this type evaporated from ingots of an 81-19 nickel-iron alloy.)

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R. F. ADAMSKY: 'Preferred Orientation and Ordering in Evaporated Films of Fe, Ni and Fe-Ni', pp. 289S–90S.

Study of the influence, on the preferred orientation of evaporated films of iron, nickel, and iron-nickel alloys, of the angle of incidence between the depositing beam and the substrate. Slight degrees of preferred orientation, in the form of (111) fibre axes, have been found for iron, nickel and 'Permalloy' films deposited on heated substrates. Annealing experiments have demonstrated the presence of superlattice ordering in 'Permalloy' films held for periods at 200°C.

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M. S. COHEN, E. E. HUBER, G. P. WEISS and D. O. SMITH: 'Investigations into the Origin of Anisotropy in Oblique-Incidence Films', pp. 291S–2S.

The data presented show that the magnetic anisotropy of oblique-incidence 'Permalloy' and iron films is not caused by an inclined texture axis or anisotropic strain. Magnetic anisotropy was observed in 'Permalloy' films deposited, at normal incidence, on non-magnetic metal films deposited at oblique incidence, a finding which is considered to suggest an agglomeration mechanism. Some indication was found that oxygen may play a rôle in oblique-incidence magnetic anisotropy.

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D. O. SMITH, E. E. HUBER, M. S. COHEN and G. P. WEISS: 'Anisotropy and Inversion in 'Permalloy' Films', pp. 295S–7S.

The authors propose a phenomenological model of field-induced and oblique-incidence anisotropy in 'Permalloy' films which relates anisotropy to inversion. They outline experimental results which either support, or are at least consistent with, the model.

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N. C. FORD: 'Domain-Wall Velocities in Thin Iron-Nickel Films', pp. 300S–1S.

The application of an apparatus, based on the Kerr magneto-optical effect, to the measurement of velocity of domain walls in thin iron-nickel films is described.

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S. METHFESSEL, S. MIDDELHOEK and H. THOMAS: 'Domain Walls in Thin Magnetic Ni-Fe Films', pp. 302S–4S.

The authors consider, in terms of Néel's proposal of a transition from the normal type of domain

wall to the Néel type at a certain film thickness (due to the thickness dependence of the magnetostatic energy), the occurrence of a cross-tie structure in nickel-iron wedge-shaped films in the thickness range 400–900Å.

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O. W. MUCKENHIRN, A. E. LABONTE and P. J. BESSER: 'Annular Uniaxially Magnetized Domains in Thin Ni-Fe Films', pp. 304S–6S.

The paper comprises a preliminary report on the magnetic behaviour of thin circular nickel-iron films exhibiting uniaxially magnetized domains with concentric circular boundaries.

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H. RUBINSTEIN and R. J. SPAIN: 'Cross-Tie Walls in Thin Films', pp. 306S–7S.

The properties of cross-tie walls are reviewed, and several new Bitter-pattern experiments dealing with cross-tie walls in thin nickel-iron films are described.

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H. SATO: 'Analysis of Magnetic Interactions in Alloys of Platinum with Iron-Group Transition Elements', pp. 327S–9S.

The magnetic properties of alloys of platinum with iron-group transition elements are analyzed and interpreted on the basis of an Ising model of ferromagnetism. In the wide ranges of composition of the face-centred-cubic alloys of the Ni-Pt, Co-Pt, Fe-Pt, Mn-Pt and Cr-Pt systems, it was found, *inter alia*, that the dependence of Curie temperature and saturation magnetization on concentration of magnetic atoms, or on the formation of superlattices, changes systematically with atomic number.

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E. I. KONDORSKY and V. L. SEDOV: 'Antiferromagnetism of Iron in Face-Centered Crystalline Lattice and the Causes of Anomalies in 'Invar' Physical Properties', pp. 331S–5S.

The anomalies in the electrical and magnetic properties of 'Invar' alloys at low temperatures, and the fact that these anomalies are observed in iron-nickel alloys with nickel contents ranging from 30 to 40 per cent., are explained in terms of an assumption that the exchange integral of electrons of neighbouring ions of iron in a face-centred lattice is negative, entailing a 'latent' antiferromagnetism in the 'Invar' alloys. The paper includes the results of investigations of the influence of pressure on the magnetic saturation and the residual resistivity of iron, nickel and iron-nickel alloys at low temperatures.

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S. A. MARSHALL and A. R. REINBERG: 'Paramagnetic Resonance Absorption of Nickel in Sapphire', pp. 336S–7S.

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R. B. FLIPPEN and S. A. FRIEDBERG: 'Magnetic Susceptibilities of Single-Crystal $NiCl_2.6H_2O$ and $CoCl_2.6H_2O$ at Low Temperatures', pp. 338S–9S.

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J. B. GOODENOUGH, A. WOLD and R. J. ARNOTT: 'Interpretation of the Magnetic and Crystallographic Properties of Several Iron, Nickel and Iron-Nickel Nitrides', pp. 342S–3S.

The data discussed were derived from a study of the saturation moment (at 4°K.) and the structure (at room temperature) of the following nitrides: $Fe_{4-x}Ni_xN$ with $0 \leqslant x \leqslant 3$ ($Fe_4N$-type structure); tetragonal, ordered FeNiN; hexagonal $Fe_{3-y}N$ with $0 < y < 1$; hexagonal $Ni_3N_{1.16}$; and orthorhombic $Fe_2N$.

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W. L. ROTH and G. A. SLACK: 'Antiferromagnetic Structure and Domains in Single Crystal NiO', pp. 352S–3S.

The study is considered to confirm the correctness of the ferromagnetic sheet structure previously proposed (in which the atomic moments lie in (111) planes).

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H. A. ALPERIN: 'Neutron-Diffraction Investigation of the Magnetic Structure of Nickel Oxide', pp. 354S–5S.

The results indicate the structure to have a single magnetic axis lying in a (111) plane, but within this plane there are domains such that the directions of the moments of these domains are distributed symmetrically.

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F. A. HAMES: 'Ferromagnetic-Alloy Phases Near the Compositions $Ni_2MnIn$, $Ni_2MnGa$, $Co_2MnGa$, $Pd_2MnSb$ and PdMnSb', pp. 370S–1S.

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E. SCHLÖMAN, J. J. GREEN and U. MILANO: 'Recent Developments in Ferromagnetic Resonance at High Power Levels', pp. 386S–95S.

The review includes two sections reporting experimental data on nickel-cobalt and nickel-zinc ferrites.

**Induced Magnetic Anisotropy in Evaporated Nickel-Iron Films**

M. PRUTTON and E. M. BRADLEY: 'The Induced Magnetic Anisotropy in Evaporated Films of Nickel-Iron Alloys.'

*Proc. Physical Soc.*, 1960, vol. 75, Apr. 1, pp. 557–74.

The factors involved in the 'Permalloy Problem' (that polycrystalline thin films of nickel-iron alloys falling within the composition range of the 'Permalloy' series exhibit a preferred direction of magnetization along a magnetic field applied during evaporation) are indicated in the introductory section of the paper by a review of the theories advanced to account for the phenomenon. A simple model (incorporating the sum of the effects of iron-pair directional ordering and a constant linear stress lying along the annealing field) is then outlined in explanation of the induced magnetic

anisotropy. The remainder of the paper is concerned with presentation and discussion of data obtained in experiments on 1500-Å-thick nickel-iron films (containing 80–85 per cent. nickel) carried out to investigate the validity of the model. The experimental technique used to prepare the films studied is described in detail.

The values for the anisotropy field *Hk*, derived from hysteresis loops measured at 400 c/s, are compared with the values expected on the basis of the simple model. The experimental observation that the minimum in anisotropy energy shifts to a lower nickel content as the substrate temperature is raised is reflected in the model, and, when the complicating effects of fibre growth are included, the major features of the observations are found to agree quite well with the model. The behaviour of the induced anisotropy is, however, shown to be complex, and consideration is given to the possibility that imperfection-ordering effects are involved.

**Influence of Plastic Deformation on the Coercive Force of Nickel-Iron Alloys**

See abstract on p. 189.

**Thermodynamic Properties of Dilute Solutions of Sulphur in Liquid Nickel-Iron Alloys**

See abstract on p. 189.

**Electrodeposition and Properties of Nickel-Iron Films**

I. TSU: 'The Preparation and Properties of Iron-Nickel Films.'

*Plating*, 1960, vol. 47, June, pp. 632–3.

Nickel-iron ferromagnetic films prepared by electrodeposition (from a solution described by WOLF and MCCONNELL in 43*rd Ann. Tech. Proceedings, Amer. Electroplaters' Soc.*, 1956, pp. 215–8) exhibit, at a nickel content of 50–80 per cent., hysteresis loops characterized by pronounced uniaxial anisotropy; at a nickel content greater than 80 per cent. such films are less oriented and have high coercive forces. In view of the dependence of properties on film composition, it was deemed of value to extend the investigation of such films to those containing less than 50 per cent. nickel. The results of the study carried out in this connexion are presented by the author.

In view of the unsatisfactoriness of Wolf's solution in exploratory experiments, a plating solution containing chloride salts of nickel and iron and a supporting electrolyte was developed. A typical solution composition is given as:

| | g./L. |
|---|---|
| $FeCl_2.4H_2O$ .. .. .. | 300 |
| $NiCl_2.6H_2O$ .. .. .. | 40 |
| $CaCl_2$ (supporting electrolyte) .. | 180 |
| HCl to control bath pH | |

With an $Fe^{++}/Ni^{++}$ ratio of 8·5, a film of 92 per cent. iron and 8 per cent. nickel was obtained using the following plating conditions:

| | |
|---|---|
| pH | 0·9–1·5 |
| Current density | 3·5 amp./dm.$^2$ |
| Temperature | 65°–90°C. |

It is stated that addition agents exert an extremely pronounced influence on the structural and magnetic properties of these films: the addition of 0·2 g./L. of thiourea will, for example, produce a 95–5 nickel iron film, even though the plating conditions are exactly the same as those used in depositing the iron-rich films.

Investigation of the magnetic properties of the iron-rich films electrodeposited showed that they are isotropic, becoming more anisotropic as the nickel-content is raised.

The magnetic properties of nickel-rich (95 per cent.) films deposited from the chloride solution differed from those produced from Wolf's bath: the former showed a high degree of anisotropy and low $H_c$; the latter were characterized by high $H_c$ and negligible anisotropy. The anisotropy of the iron-nickel film does not, it is therefore concluded, depend entirely on the composition of the alloy.

## CAST IRON

**Properties and Applications of 'S-G Ni-Resist' Austenitic Cast Irons**

A. B. EVEREST and O. NICKEL: 'Spheroidal-Graphite Types of 'Ni-Resist' Cast Iron.'

Translation of a paper published in *Konstruktion*, 1959, vol. 11, No. 10, pp. 390–5.

MOND NICKEL COMPANY, LTD., *Publn.* 2052, 1960; 8 pp.*

*Reprint* from *Foundry Trade Jnl.*, 1960, vol. 108, Apr. 28, pp. 515–22: see abstract in *Nickel Bulletin*, 1960, vol. 33, No. 6, pp. 141–2.

**High-Temperature Fatigue Strength of Nickel-containing Cast Irons**

R. P. FELGAR: 'Fatigue Tests on Three Cast Irons at Elevated Temperatures.'

*Proc. Amer. Soc. Testing Materials*, 1959, vol. 59, pp. 767–73.

The author reports data derived from axial-stress fatigue tests carried out to determine the fatigue strength (for a life of $10^7$ cycles) of a nickel-molybdenum iron, a nickel-containing nodular (spheroidal-graphite) iron and 'Meehanite' cast iron at temperatures in the range 750°–1000°F. (400°–540°C.).

Pulsating fatigue tests (zero to maximum stress) were performed on all three materials, and completely-reversed push-pull tests were conducted on the nickel-molybdenum and nodular irons. The staircase method of fatigue testing was employed, and the data obtained were subjected to statistical analysis.

Within the limits of the temperature range studied, none of the materials exhibited a significant variation in fatigue strength with temperature. At $10^7$ cycles the pulsating fatigue strength of 'Meehanite' was

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found to be essentially constant from 750° to 1000°F., and, in the case of the nickel-molybdenum iron, fatigue strength varied little over the range 900°–1000°F. Nodular iron was considerably stronger in pulsating fatigue at 900°F. (480°C.) than either 'Meehanite' or the nickel-molybdenum iron. The nickel-molybdenum iron was only slightly stronger than the 'Meehanite'.

Mean stress had a significant effect on the alternating fatigue strength of both the nodular and nickel-molybdenum irons at 900°F. Their alternating fatigue strengths were roughly 30 per cent. less for pulsating stress than for completely reversed stress.

### Influence of Nickel and Silicon on the Eutectic Carbon Content of S.G. Iron

R. D. SCHELLENG: 'Influence of Nickel up to 37 per cent. and Silicon up to 3 per cent. on the Eutectic Carbon Content of Cast Iron.'

*Modern Castings*, 1960, vol. 37, June, pp. 87–9.

Austenitic cast irons, containing 20–35 per cent. nickel and treated with magnesium to produce a spheroidal-graphite structure, are being used increasingly for such components as pumps, valves, exhaust manifolds, turbocharger, casings, etc. (see *Nickel Bulletin*, 1960, vol. 33, No. 6, pp. 141–2). That silicon decreases the eutectic carbon content of iron is well known, but quantitative data on the combined effects of silicon and nickel (information essential for proper foundry control) are sparse. The investigation described by the authors was therefore conducted to study the effects, on the eutectic carbon content of spheroidal-graphite cast iron, of nickel contents up to 37 per cent. and silicon contents up to 3 per cent.

The data presented demonstrate that the eutectic carbon content of iron decreases approximately linearly with increase in nickel content up to about 37 per cent., and (if the accepted value of 4·3 per cent. carbon in the absence of nickel is included in the data) the rate of decrease is found to be 0·047 per cent. carbon/per cent. nickel.

Study of three series of melts of approximately constant nickel content (20, 30 and 34 per cent.), but varying in silicon content, showed that the effect of silicon on the eutectic carbon content was essentially linear at each particular nickel level, though the magnitude of the effect varied with the nickel content. At 20 per cent. nickel, the eutectic carbon content decreased at the rate of 0·22 per cent. for each per cent. of silicon added; at 34 per cent. nickel, the rate of decrease was 0·14 per cent.; at 30 per cent. nickel, the rate of decrease was 0·19 per cent. for each per cent. silicon.

The observed effects of nickel and silicon on the eutectic carbon content may, it is stated, be expressed by the following formula:

$$C_{eutectic} = 4{\cdot}30 - 0{\cdot}33\ (\%\ Si) - 0{\cdot}047\ (\%\ Ni) + 0{\cdot}0055\ (\%Ni)\ (\%\ Si).$$

The formula is valid only for nickel contents up to 37 per cent. and silicon contents up to 3 per cent. The 95 per cent. confidence limit (twice the standard error of estimate) is ±0·16 per cent. carbon.

### Influence of Composition on the Notch Toughness of S.G. Iron

S. V. ARNOLD: 'Notch Toughness of Ductile Cast Iron as a Function of Composition.'

*Watertown Arsenal, Report TR* 330/17, undated; 7 pp.+ tables and figures.

In the investigation reported, the Charpy V-notch impact strength of specimens of ductile (spheroidal-graphite) iron were determined, in the as-cast and annealed conditions, as a function of temperature. The composition of the specimens studied was varied so as to throw light on the influence, on notch toughness, of the following alloying additions: carbon, silicon, manganese, phosphorus, magnesium, nickel, nickel+manganese, nickel+manganese+chromium, molybdenum, vanadium, copper, boron, cerium.

The data presented indicate that, of the alloying elements studied, only silicon had a beneficial effect on toughness: the silicon contents resulting in maximum toughness in the as-cast and annealed conditions were, respectively, 3·17 and 2·15 per cent. The results also show that, in annealed S.G. iron, higher tensile properties can be attained, with least detriment to toughness, by addition of nickel.

The best of the as-cast irons exhibited transition temperatures (defined as the minimum temperature compatible with the achievement of 10 ft.-lb. impact strength) in the region of 60°C.; transition temperatures in the best of the annealed irons approached −20°C.

### Behaviour of S.G. Iron under Compressive Stress

P. K. TROJAN, R. A. FLINN and D. J. REESE: 'Behavior of Ductile Cast Iron Under Compressive Stress.'

*Proc. Amer. Soc. Testing Materials*, 1959, vol. 59, pp. 922–9.

Little data are available on the compressive characteristics of irons and steels, and, in many applications, hardness and tensile properties have perforce been used as an indication of service performance under compressive loading. In this paper the authors give details of a test programme intended to gain information on the behaviour, under compressive stress, of a spheroidal-graphite iron containing carbon 3·6, silicon 2·25, manganese 0·54, nickel 1·04, magnesium 0·072, per cent.

Compression specimens machined from ½-, 1- and 2-in. (1·25-, 2·5- and 5-cm.) 'Y' blocks were austenitized, quenched from 1600°F. (870°C.), and tempered 1 hour at 1300°, 1150°, 925° and 300°F. (705°, 620°, 495°, 150°C.).

Compression yield strength (0·1 per cent. offset) and Brinell hardness ranged from 66,000 p.s.i. (29·5 t.s.i.: 46·5 kg./mm.²) to 215,000 p.s.i. (96 t.s.i.: 151 kg./mm.²) and 215 to 585 B.H.N., respectively. Within this range, the maximum deviation between the three section sizes was only ±4000 p.s.i. and ±8 B.H.N. The compression modulus did, however, indicate some sensitivity to the presence of less perfect

graphite spheroids in the 1-in. and 2-in. sections: the average modulus determined was $24 \times 10^6$ p.s.i., but the modulus of larger sections was $4 \times 10^6$ p.s.i. lower than this value at low hardness levels, and $2 \times 10^6$ p.s.i. lower at higher hardness.

## CONSTRUCTIONAL STEELS

### Upper- and Lower-Bainite Formation in a Nickel-Chromium Steel

J. S. WHITE and W. S. OWEN: 'Kinetic Features of Upper- and Lower-Bainite Formation in a Plain-Carbon and a 3% Ni-Cr Steel.'

*Jnl. Iron and Steel Inst.*, 1960, vol. 195, May, pp. 79–82.

Previous work having shown that upper and lower bainite form in high-purity iron-carbon alloys and plain-carbon steels by kinetically different processes, the present work (on a 3 per cent. nickel-chromium steel and a eutectoid plain-carbon steel) was carried out as a preliminary to the determination of the extent to which the basic features revealed by those studies occur in bainitic transformations in low-alloy steels.

A high-precision technique was developed to enable the isothermal decomposition of austenite to be followed by means of continuous electrical-resistivity measurements.

The data presented demonstrate that the general features of the bainite reaction in the 3 per cent. nickel-chromium steel are similar to those in plain-carbon steels. In both steels the transition from upper to lower bainite (as reflected in the change in activation energy) occurs at about 350°C., a finding which indicates that the temperature range over which lower bainite is formed in the alloy steels is small (320°–350°C.). The presence of the alloying elements in the 3 per cent. nickel-chromium steel did not significantly affect the activation energy for upper-bainite formation.

The lower-bainite reaction was found to occur over an appreciably wider range than that over which the lower C-curve is detectable. It is tentatively suggested that, on passing into the lower-bainite range, the morphological change and the appearance of ε-carbide do not coincide, a factor which is deemed to warrant further investigation.

### Mechanical Properties of Nickel-Alloy Steels at Sub-Zero Temperatures

MOND NICKEL COMPANY, LTD.: 'The Mechanical Properties of Nickel-Alloy Steels at Sub-Zero Temperatures.'*

*Publn.* 1648, 1960; 80 pp.

In the engineering, aircraft and chemical industries, alloy steels are frequently subjected to stresses at sub-zero temperatures, and there is increasing need for information on the mechanical properties of standard steels at these temperatures. This publication presents the results of a survey conducted (in the Laboratory of the Development and Research Department of The Mond Nickel Company, Ltd.) to obtain data on the tensile and impact properties of representative commercially available nickel-containing steels at temperatures ranging from +20°C. to −196°C.

The publication is in two sections. In the first, and introductory, section consideration is given to the following factors and their effects on the properties of steels at low temperatures: temperature, presence of notches, size and rate of application of the stress, deoxidation and grain-size, microstructure, composition.

The second, and main, section of the publication contains data on the tensile, hardness and impact properties of the types of steel listed below. Test specimens were obtained from plate and/or bar stock, and, in some cases, tests were carried out also on welded specimens. Full particulars are given of the composition of the steels and the heat-treatments to which they were subjected.

*Direct-Hardening Steels*

| | |
|---|---|
| B.S. En 23 | 3% Ni-Cr |
| B.S. En 24 | 1½% Ni-Cr-Mo |
| B.S. En 25 | 2½% Ni-Cr-Mo (Medium-Carbon) |
| B.S. En 26 | 2½% Ni-Cr-Mo (High-Carbon) |
| B.S. En 28 | 3½% Ni-Cr-Mo |
| B.S. En 30B | 4¼% Ni-Cr-Mo |
| B.A.C.E. 165 | 1¾% Ni-Cr-Mo-V |

*Case-Hardening Steels*

| | |
|---|---|
| B.S. En 33 | 3% Ni |
| B.S. En 34 | 2% Ni-Mo (lower-carbon) |
| B.S. En 36A, B.S. En 36B | 3% Ni-Cr |
| B.S. En 37 | 5% Ni |
| B.S. En 39A | 4¼% Ni-Cr |
| B.S. En 39B | 4¼% Ni-Cr-Mo |

*Special Steel for Low Temperatures*

| | |
|---|---|
| A.S.T.M. A353 | 9% Ni (low-carbon) |

*Martensitic Rust-Resisting Steel*

| | |
|---|---|
| B.S. En 57 | 16% Cr-2% Ni |

*Austenitic Rust-, Acid- and Heat-Resisting Steels*

| | |
|---|---|
| B.S. En 58A | 18% Cr-8% Ni |
| B.S. En 58B | 18% Cr-8% Ni, Ti-stabilized |
| B.S. En 58F, B.S. En 58G | 18% Cr-10% Ni, Nb-stabilized |
| B.S. En 58J | 18% Cr-8% Ni-3% Mo, Ti-stabilized |
| — | 25% Cr-20% Ni |

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In the case of the ferritic steels, tests were made over a range of temperatures spanning the transition from tough to brittle fracture; the properties of the austenitic specimens were determined over the temperature range +100°C. to −196°C. Hardness determinations were made at room temperature, −78°C. and −196°C.

**High-Strength Steels: Properties, Heat-Treatment and Forming Practices**

See abstract on p. 217.

**Sharp-Edge-Notch Tensile Strength of Nickel-containing High-Strength-Steel Sheet**

See abstract on p. 215.

**Influence of Non-Metallic Inclusions on the Fatigue Properties of High-Strength Steels**

M. ATKINSON: 'The Influence of Non-Metallic Inclusions on the Fatigue Properties of Ultra-High-Tensile Steels.'

*Jnl. Iron and Steel Inst.*, 1960, vol. 195, May, pp. 64–75.

Although the presence of inclusions is assumed to be detrimental to the mechanical properties of steel, there has been little evidence of any direct relationship between non-metallic content and fatigue properties. An attempt to correlate these two factors is described in the present paper.

In preliminary work specimens of five low-alloy nickel-chromium – molybdenum – (vanadium) steels were heat-treated to a tensile strength of about 125 tons/sq. in. (280,000 p.s.i.: 197 kg./mm.$^2$) and then subjected to fatigue testing. The non-metallic content, as assessed by the Fox inclusion count, appeared to bear no relationship to the fatigue properties of the samples examined. Consideration of alternative forms of counting technique led to the development of a procedure (the 'Fairey inclusion count') which, based on the stress concentration attributable to the observed sections of individual inclusions, permits assessment of the effect of particle shape. Assessment is facilitated by reference to a chart relating geometric shapes to their calculated values. In view of the problems associated with the metallographic determination of particle shape and size, other factors which may contribute to the stress concentration in the neighbourhood of a non-metallic inclusion were discounted.

The new inclusion count is described in the paper. Its validity was determined experimentally by direct comparison of the non-metallic content and fatigue-resistance of the five steels: the test results are summarized.

This correlation indicated that inclusions have a direct influence on the fatigue-resistance of steels hardened to ultra-high-strength levels, and it is assumed, therefore, that the Fairey inclusion count measures some relevant quality of the inclusions, which appears to be a function of stress concentration and decrease in load-bearing area. (The count value is thought to be related to the probable stress concentration due to the inclusions at any point in the metal.) This finding suggests that the theory proposed in explanation of the effect of inclusions on the fatigue mechanism is basically correct: i.e., that tensile strength is incompletely transduced by the foreign particles, and the stress is concentrated in the surrounding metal in accordance with the shape of the cavities they occupy.

**Influence of Elevated-Temperature Exposure on Nickel-containing Pressure-Vessel Steels**

A. W. PENSE, J. H. GROSS and R. D. STOUT: 'Effect of Elevated-Temperature Exposure on Heavy-Section Pressure-Vessel Steels.'

*Welding Journal*, vol. 39, June, pp. 231s–5s.

In recent years, investigations have been in progress in the U.S.A. to examine the feasibility and advantages of using high-strength steels for pressure-vessel construction. Previous studies had demonstrated the general suitability of such steels for use, at ambient temperatures, in conventional thicknesses and also in heavy sections, particularly if the latter are spray-quenched and stress-relieved (see, for example, paper by RUBIN, GROSS and STOUT abstracted in *Nickel Bulletin*, 1959, vol. 32, No. 6, pp. 184–5). Little or no information was, however, available on the behaviour of the steels under the conditions obtaining in boilers and pressure vessels operating at elevated temperatures: in particular, data were required on tensile properties (which might be influential during long-term operation at elevated temperatures) and changes in notch toughness (which may influence safety during shut-down operations). The investigation now described was initiated to obtain the required information.

The compositions of the two carbon and seven low-alloy high-strength steels selected for study are given in the table on p. 206.

Prior to stress relief, all the specimens were subjected to heat-treatments intended to reproduce the structure typical of either a normalized 4-in. (10-cm.) plate or of a point near the surface of a spray-quenched plate of the same thickness. Some specimens were then strained 5 per cent. and re-stress-relieved, but (with the exception of those strained 5 per cent. and then aged for various times at room temperature) all the specimens were exposed at 500° and 700°F. (260° and 370°C.) for 2,000, 8,000 or 16,000 hours. After exposure, tensile properties and notch ductility were determined.

No significant embrittlement (greater than a 25F.° (14C.°) increase in transition temperature) occurred in any of the steels after ageing at room temperature or 500°F. (260°C.) for times up to 16,000 hours. After ageing at 700°F. (370°C.), however, certain of the alloy steels (i.e., 'T-1', 'HY-80', 'HY-65' and 'A203') exhibited embrittlement (40–100F.° increase in the transition temperature). Maximum embrittlement, which occurred at about 8,000 hours at 700°F., appeared to be independent of the condition of the steel. After 16,000 hours at 700°F.

**Composition of Pressure-Vessel Steels**

(*See abstract on p. 205*)

| Steel | C % | Mn % | P % | S % | Si % | Ni % | Cr % | Mo % | V % | Ti % | Cu % | B % |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| A.S.T.M. A285 .. | 0·20 | 0·53 | 0·020 | 0·016 | — | — | — | — | — | — | — | — |
| 48s5 .. .. | 0·15 | 1·09 | 0·033 | 0·028 | 0·18 | — | — | — | 0·09 | 0·009 | — | — |
| A.S.T.M. A212 .. | 0·28 | 0·70 | 0·010 | 0·021 | 0·24 | — | — | — | — | — | — | — |
| A.S.T.M. A203 .. | 0·15 | 0·64 | 0·010 | 0·026 | 0·22 | 3·64 | — | — | — | — | — | — |
| 'HY-65' .. .. | 0·12 | 0·48 | 0·013 | 0·032 | 0·21 | 2·16 | — | 0·39 | — | — | 0·70 | — |
| A.S.T.M. A302 .. | 0·20 | 1·32 | 0·022 | 0·030 | 0·25 | — | — | 0·42 | — | — | — | — |
| A.S.T.M. A387 .. | 0·09 | 0·44 | 0·013 | 0·011 | 0·25 | — | 2·18 | 0·96 | — | — | — | — |
| 'T-1' .. .. | 0·15 | 0·93 | 0·015 | 0·022 | 0·27 | 0·89 | 0·48 | 0·44 | 0·06 | — | — | 0·0031 |
| 'HY-80' .. .. | 0·15 | 0·28 | 0·016 | 0·015 | 0·19 | 2·86 | 1·72 | 0·50 | — | — | — | — |

the stress-relieved steels exhibited the same degree of embrittlement as after 8,000 hours, whereas the strained and re-stress-relieved specimens showed a tendency to recover from the maximum embrittlement. The 700°F. embrittlement was accompanied by a corresponding increase in the tensile strength.

The results obtained did not suggest a specific mechanism by which the embrittlement occurs, but it is noted that only the nickel-containing steels were embrittled.

### Submerged-Arc Welding of 'Hy-80' Steel

W. J. LEWIS, G. E. FAULKNER, D. C. MARTIN and P. J. RIEPPEL: 'Submerged-Arc Welding 'HY-80' Steel.' *Welding Jnl.*, 1960, vol. 39, June, pp. 266s-72s.

One of the prime considerations in the construction of a submarine hull is that it should withstand severe impact at temperatures in the region of 30°F. (−1°C.). Most of the hulls of U.S. submarines are fabricated from the low-alloy nickel-chromium-molybdenum steel 'HY-80', using standard low-hydrogen welding electrodes and pre-heating operations. Although many of the joints involved in the construction of the hull lend themselves to automatic-welding techniques, the properties of weldments deposited by the submerged-arc process are not satisfactory for applications involved in submarine construction. The study reported was conducted to develop methods suitable for producing, by the submerged-arc process, welds which would withstand severe impact loading at low temperatures. The specific aims were to obtain submerged-arc weld metals with yield strengths of at least 80,000 p.s.i. (35·5 t.s.i.: 56 kg./mm.$^2$) and Charpy V-notch toughness of 20 ft.-lb. (2·76 kgm.) at −100°F. (−73°C.) or nil-ductility temperature values of −100°F. in both the as-welded and stress-relieved conditions. The authors outline in the paper the results obtained in an evaluation of several experimental filler wires. Data on the effects of nickel, manganese and molybdenum on weld-metal notch toughness and tensile properties are discussed, and a comparison is made of the properties of submerged-arc welds and inert-gas-shielded metal-arc welds made with the same filler wire under similar conditions.

The compositions of the nine experimental filler wires studied enabled the effects of alloying elements to be determined over the following ranges: nickel 1·4-3, manganese 1·2-1·8, molybdenum 0·05-0·8, per cent.

The authors' conclusions are quoted below.

'1. Increasing nickel content lowered weld-metal notch toughness, especially at testing temperatures above −40°F. (−40°C.). However, at testing temperatures below −40°F., increased nickel appeared to be beneficial.

'2. Increasing manganese content also lowered weld-metal notch toughness at testing temperatures above −40°F., but, at testing temperatures below −40°F., the notch toughness of the welds was about the same.

'3. Increasing molybdenum content generally lowered weld-metal notch toughness.

'4. The tensile and yield strengths of the weld metals were improved by increasing the nickel, manganese or molybdenum contents. However, the improvement in strength obtained by increasing the manganese or molybdenum contents was greater than that obtained by increasing the nickel content.

'5. The notch toughness of the weld metals was improved, and the tensile and yield strengths were lowered, by stress-relieving operations.

'Studies to find the differences between submerged-arc and inert-gas-shielded metal-arc weld metals showed the following:

'1. The notch toughness of weld metals made by the inert-gas-shielded metal-arc process was higher than the notch toughness of weld metals made by the submerged-arc process.

'2. The difference found in the notch toughness of weld metals made by the two processes was attributed to the number of inclusions, oxygen content and silicon content.

'3. The tensile properties of weld metals made by the two processes were practically the same.'

## HEAT- AND CORROSION-RESISTING MATERIALS

### Extrapolation of Creep-Rupture Data

A. MENDELSON and S. S. MANSON: 'The Extrapolation of Families of Curves by Recurrence Relations, with Applications to Creep-Rupture Data.'

*Amer. Soc. Mechanical Engineers*, 1959, *Paper* 59-*A*-155; 8 pp.

The author outlines a method which is claimed to enable direct extrapolation of a family of curves (e.g., creep-rupture) without the assumption of any particular parametric form. The method 'is based on the assumption that a family of curves, such as a set of isothermal curves, can be represented approximately by a finite-difference recurrence relation. Once the coefficients of this recurrence relation are determined, it is a simple matter to extrapolate each member of the family individually. The method has the advantage that it does not require an explicit knowledge or assumption as to the analytic character of the curves, or as to what parametric form to use to correlate the data, although implicitly it assumes any of the broad class of functions which satisfy linear finite-difference equations. It is thus much more general in nature than the parametric methods previously discussed, and can be used by itself to extrapolate creep-rupture data or as an independent check on any of the parameter methods. In addition, the method treats the data in such a way as to give more weight to the higher time data than to the lower time data.'

The use of the technique is exemplified in the paper, and the results of its application to the extrapolation of creep-rupture data for the following materials are presented: 'S-590', 'Nimonic 80A', 'A-286', carbon steel, chromium stainless steel, 18-8 chromium-nickel stainless steel.

### Kinetics of Softening of Chromium-Nickel Alloys during Annealing

M. P. ARBUZOV and M. P. KRULIKOVSKAYA: 'Kinetics of Dehardening of Chromium-Nickel Alloys.'

*Fiz. Metal. Metalloved.*, 1959, vol. 7, No. 3, pp. 432-7. Translated in *Physics of Metals and Metallography*, vol. 7, No. 3, pp. 106-10.

Chromium-nickel alloys containing 3·87, 7·65 and 14·43 per cent. chromium were hardened by plastic deformation and then annealed at 550°, 600°, 650° and (in the case of the third alloy) at 700°C. Changes in hardness, the second order lattice defects $\Delta a/a$, and in the dimensions of the mosaic blocks $D$ were determined as a function of the annealing time, and the kinetic curves obtained were correlated with the chromium content of the alloys. The activation energy associated with the removal of the second-order defects was calculated.

The data presented are regarded as indicating that the hardening and softening processes in a solid solution are related to changes in crystal microstructure. The activation energy for the removal of the lattice defects $\Delta a/a$ is shown to increase linearly with increase in chromium content.

*See also*

### Influence of Aluminium and Titanium on the Hardening and Softening of Nickel-Chromium Alloys

M. P. ARBUZOV and V. G. CHERNYI: 'Effect of Aluminium and Titanium on the Hardening and Dehardening of Alloys of the Chrome-Nickel Type.'

*Fiz. Metal. Metalloved.*, 1959, vol. 7, No. 3, pp. 438-42. Translated in *Physics of Metals and Metallography*, vol. 7, No. 3, pp. 111-15.

Studies of the hardening and softening mechanisms in nickel-base alloys having demonstrated that the presence of chromium facilitates hardening of the alloy during deformation, and the preservation of the hardened state to higher annealing temperatures (*see previous abstract*), the investigation now reported was undertaken to determine the influence of aluminium and/or titanium on changes in the crystal structure (second-order defects and mosaic-block dimensions) involved in hardening and softening nickel-chromium alloys.

The five alloys studied, two of which were age-hardenable, contained carbon 0·025-0·04, chromium 19·55-19·67, titanium 0-2·3, aluminium 0·16-2, per cent. After homogenization, specimens were subjected to 80 per cent. plastic deformation and then annealed in the range 400°-800°C. Hardness measurements were made and the block size and the size of second-order defects were determined by X-ray line-width techniques.

Addition of aluminium and titanium results it is concluded, in a greater degree of hardening after deformation and also shifts the softening processes to higher temperatures, effects which the authors attribute to the tendency of aluminium and titanium to increase the inter-atomic bonds in the solid-solution lattice.

### Metallographic Technique for Study of Phase Transformation in Nickel-Titanium Alloys

R. P. MORENSKI and N. S. PITEA: 'Metallographic Techniques for Age Hardening High-Temperature Alloys.'

*Metal Progress*, 1960, vol. 77, Mar., pp. 99-100.

Control of the high-temperature properties of nickel-base alloys rendered age-hardenable by titanium additions has been greatly facilitated by metallographic studies of phase changes during ageing. In

such alloys creep-resistance is governed by microstructure: the transformation of $Ni_3Ti$, the age-hardening constituent, from the face-centred-cubic form to the close-packed-hexagonal form occurs within the grains and at the grain boundaries, and results in over-ageing. Study of these critical changes is of obvious importance. In this short article the authors outline the metallographic techniques evolved to reveal changes in the microstructure of an alloy containing 87·8 per cent. nickel and 12·2 per cent. titanium, together with trace amounts of boron.

Optical and electron micrographs, superimposed on the T.T.T. curve, illustrate the transformation of $N_3Ti$ from the face-centred-cubic structure (cubic particles) to hexagonal form (lamellar and Widmanstätten structure).

### Distortions and Binding Forces in Nickel-base Solid Solutions

See abstract on p. 193.

### Studies of Oxide Films on Nickel-Chromium and Nickel-Chromium-Iron Materials

I. PFEIFFER: 'Electron-Optical Studies of Oxide Films on Nickel-Chromium and Nickel-Chromium-Iron Alloys.'

*Zeitsch. f. Metallkunde*, 1960, vol. 51, June, pp. 322–6.

On account of their high oxidation-resistance, nickel-chromium and nickel-chromium-iron materials have found wide application as electrical-resistance alloys, and, in the last 30 years, additions of cerium, calcium, aluminium or zirconium, and use of higher silicon and lower manganese contents, have achieved a considerable improvement in service life. Attempts to determine the causes of this high oxidation-resistance have shown that the presence of an adherent diffusion-inhibiting superficial oxide film is an essential factor. The investigation described, which was carried out to determine the composition of the oxide films present on currently available alloys (see compositions below), was intended to form a preliminary stage to the determination of the composition and crystal structure associated with optimum protection.

| Alloy | Ni % | Cr % | Fe % | Mn % | Si % |
|---|---|---|---|---|---|
| 80-20 .. | 76·4 | 21 | 1 | 0·2 | 1·4 |
| 60-15 .. | 62 | 18 | 18·3 | 0·5 | 1·2 |
| 30-20 .. | 33 | 21 | 42·9 | 0·8 | 2·3 |

Specimens of each alloy were heated to produce surface oxide films 300–500Å and 10–30μ thick, and then subjected to electron-microscopical examination. The data on the films 10–30μ thick were supplemented by the results of chemical analysis.

The compositional differences determined between the thinner and thicker oxide films are regarded of interest in relation to the oxidation mechanism. Films in the 300–500Å range consisted predominantly of spinels; the thicker films contained a considerable amount of $Cr_2O_3$. These differences are discussed from the point of view of selective oxidation.

### Mechanism of Protective-Scale Formation on Nickel-containing Copper-base Alloys

See abstract on p. 194.

### Temperature-Dependence of the Shear Modulus and Internal Friction of Nickel-base Alloys

I. G. POLOTSKII and Z. L. KHODOV: 'The Dependence on Temperature of the Shear Modulus and Internal Friction of some Nickel-based Alloys.'

*Fiz. Metal. Metalloved.*, 1959, vol. 7, No. 2, pp. 274–7. Translated in *Physics of Metals and Metallography*, Vol. 7, No. 2, pp. 110-13.

A study was made (using the torsional oscillation method) of the variation, as a function of temperature over the range room temperature to 800°C., of the shear modulus and internal friction of nickel-molybdenum alloys containing 4·98, 9·85 and 14·9 per cent. of molybdenum, a nickel-chromium-aluminium alloy containing 19·78 per cent. of chromium and 2·2 per cent. of aluminium, and a nickel-chromium-titanium alloy containing 19·58 per cent. of chromium and 2·5 per cent. of titanium. The specimens were studied in the annealed condition and measurements were made *in vacuo*.

In the case of all the alloys studied, the temperature/shear-modulus curve deviated from linearity in the same range of temperature as that in which a sharp increase in internal friction occurred. The Poisson coefficient calculated for the nickel-chromium-titanium alloy increased markedly from a temperature in the range 400°–500°C., an effect which is tentatively attributed to the considerable increase in the plasticity of the alloy.

### Thermal and Electrical Conductivities of 'Nimonic' Alloys

R. W. POWELL and R. P. TYE: 'Thermal and Electrical Conductivities of Nickel-Chromium ('Nimonic') Alloys.'

*Engineer*, 1960, vol. 209, Apr. 29, pp. 729–32.

Heat conductivity, although not a property of primary consideration in the evolution of the nickel-chromium-base high-temperature alloys of the 'Nimonic' series, influences the thermal stresses developed and is accordingly of importance. In the experiments (carried out at the National Physical Laboratory) now reported, the thermal conductivity ($K$) and electrical resistivity ($\rho$) of the following alloys were determined at temperatures in the range 50°–800°C. (50°–1000°C. in the case of 'Nimonic 105'): 'Nimonic 75', 'Nimonic 80', 'Nimonic 90', 'Nimonic 95', 'Nimonic 100', 'Nimonic 105'. The experimental techniques employed are fully described.

The values tabulated are shown to conform (to within 6 per cent.) with the line

$$K=2\cdot2\times10^{-8}\,(T/\rho)+0\cdot060,$$

where $T$ is the absolute temperature, $K$ is expressed in joule cm./cm.$^2$ sec. °C., and $\rho$ in ohm cm.$^2$/cm. This equation is suggested as a means of deriving, from the relatively simple measurement of electrical resistivity, the thermal conductivity of other alloys of this type.

### Development of Improved Nickel-base High-Temperature Alloys

J. C. FRECHE, T. J. RILEY and W. J. WATERS: 'Continued Study of Advanced-Temperature Nickel-base Alloys to Investigate Vanadium Additives.'

*Nat. Aeronautics and Space Administration, Tech. Note D*-260, Feb. 1960; 34 pp.

The research reported comprised the second phase of a test programme to develop a nickel-base turbine-blading alloy capable of operating at higher temperatures than those currently available. In the initial phase of the investigation, modification of a basis composition* with small additions of boron, carbon, titanium, or titanium + carbon culminated in the development of a series of alloys with good stress-rupture properties, oxidation-resistance and impact characteristics: see *Nat. Aeronautics and Space Administration, Memorandum* 4-13-59*E*, the scope and salient findings of which were noted in *Nickel Bulletin*, 1959, vol. 32, No. 8, pp. 263–4.

In the programme now described attempts were made, using the same basis composition, to achieve greater strength at elevated temperatures by means of alloying additions of vanadium. Vanadium being a strong carbide former, it was believed that the element might also act as a strengthener in a manner analogous to titanium. Consideration was also given to the possibility of improving the properties of the alloy by the use of tungsten as a possible matrix strengthener in addition to molybdenum.

The effects of the following additions were established:

| % |
|---|
| 1·0 V + 0·125 C |
| 1·5 V + 0·125 C |
| 2·5 V + 0·125 C |
| 3·5 V + 0·125 C |
| 5·0 V + 0·125 C |
| 1·0 V + 0·25 C |
| 1·5 V + 0·25 C |
| 2·5 V + 0·25 C |
| 3·5 V + 0·25 C |
| 5·0 V + 0·25 C |
| 4 W |
| 2 W + 2·5 V + 0·125 C |
| 4 W + 2·5 V + 0·125 C |
| 6 W + 2·5 V + 0·125 C |
| 8 W + 2·5 V + 0·125 C |

The basic nickel content was decreased by a percentage equal to the carbon and vanadium additions; the tungsten addition was accounted for by an appropriate decrease in the molybdenum content.

The alloys were evaluated on the basis of stress-rupture life, impact-resistance and formability. The short-time tensile properties of the most promising compositions were determined at room temperature and at 1800°F. (980°C.). Hardness data were obtained for most of the alloys investigated. Melts were made by high-frequency induction heating under an argon blanket, and investment-casting techniques were employed to obviate the need for extensive machining of test samples.

Full particulars are given of test conditions. The data obtained are presented in detail.

The strongest vanadium/carbon modification of the basis alloy (basis alloy + 2·5 vanadium + 0·125 carbon) displayed an as-cast rupture life of 565 hours at 1800°F. (980°C.) and 15,000 p.s.i. (6·7 t.s.i.: 10·5 kg./mm.$^2$). As-cast specimens of the strongest tungsten-vanadium-carbon modification (4 tungsten + 2·5 vanadium + 0·125 carbon) exhibited, at the same stress, rupture lives of 768, 301 and 101 hours at, respectively, 1800°, 1850° and 1900°F. (980°, 1010° and 1035°C.). The impact-resistance of all the alloys is considered good (in most cases, it exceeded 62·5 in.-lb.). Both the stress-rupture and impact properties of the two strongest modifications represent substantial improvements over the strongest alloy (basis alloy + 1·5 titanium + 0·125 carbon) developed in the earlier investigation.

It is emphasized that the alloys evolved must be regarded as essentially casting alloys (although some exhibited a limited degree of workability). None of the alloys required vacuum-melting techniques: all were melted under an inert-gas blanket and were poured in air.

The vanadium-bearing alloys showed no evidence of catastrophic oxidation after prolonged stress-rupture testing in air at 1800°, 1850° and 1900°F.

### Composition/Creep-Strength Relationship in the Nickel-Chromium System

I. I. KORNILOV and N. T. DOMOTENKO: 'On the Question of the Basic Types of "Composition–Creep Strength" Diagram for Metal Systems.'

*Fiz. Metal. Metalloved.*, 1959, vol. 7, No. 3, pp. 372–7. Translated in *Physics of Metals and Metallography*, vol. 7, No. 3, pp. 49–53.

Previous investigations of the functional relationship between the creep strength of metal systems and their chemical composition, their structure and their equilibrium diagram have revealed the existence of three principal types of composition/creep-strength diagram. The second type, which includes systems with limited solid solutions extending over considerable composition ranges, and with solubilities varying with temperature, is the subject of the present paper. It has been shown that creep strength increases with increase in the concentration of the solid solution, reaching maxima at those test temperatures which correspond to the boundary between the saturation limit and the supersaturation range for the solid

* Molybdenum 8, chromium 6, aluminium 6, zirconium 1, wt. per cent., remainder nickel.

solution. As the structure becomes more heterogeneous, and as coarse particles of the excess phase are formed, the creep strength of alloys comprising the system decreases. The investigation now reported was initiated to study the composition/creep-strength relationship in nickel-chromium alloys ranging over the entire system.

Creep strength was determined at 800° and 1000°C. (at stresses of 12·3 and 2·7 kg./mm.², respectively), and the data obtained were employed in the construction of two composition/creep-strength diagrams. Examination of these diagrams reveals the presence of two creep-strength maxima, which coincide with the two saturation ranges for the nickel-rich and chromium-rich solid solutions. At 800°C. alloys in the saturation-boundary range of nickel in chromium were the more creep-resistant; at 1000°C. the nickel-rich solid solutions at the saturation boundary exhibited the higher creep strength. This reversal in creep strength with temperature is attributed to differences in the strength of the chemical bond and in the crystal lattices of the two solid solutions.

### Influence of Boron and/or Zirconium on the Creep Properties of 'Nimonic' Alloys

K. E. VOLK and A. W. FRANKLIN: 'Improvement of the Creep Properties of Heat-Resisting Nickel Alloys by Addition of Small Amounts of Boron and Zirconium.'

*Zeitsch. f. Metallkunde*, 1960, vol. 51, Mar., pp. 172–9.

It is known that trace elements can influence, either deleteriously or beneficially, the creep-resistance of metals and alloys: the adverse effects of sulphur have, for example, been recognized in the standards covering the 'Nimonic' high-temperature alloys, and the beneficial effects of boron and zirconium have been studied in other alloys by DECKER *et al.* (see *Nat. Advisory Committee for Aeronautics, Report* 1392: abstract in *Nickel Bulletin*, 1959, vol. 32, No. 12, p. 370). Although the investigations now reported were carried out independently (by Volk at the Defence Research Laboratory, Melbourne, and by Franklin at the Laboratory of The Mond Nickel Company, Ltd.), similar conclusions have been drawn from the results obtained.

The creep-resistance and structure of 'Nimonic 80A', 'Nimonic 90' and 'Nimonic 95' were studied as a function of trace additions of boron and/or zirconium. The additions were found to increase considerably the time-to-rupture of the three alloys at their optimum service temperatutes. In addition, the creep curve determined for the boron/zirconium-containing alloys differed markedly from that associated with the standard alloys: the creep-rate in the second stage of creep was less, and the start of the third stage was shifted to longer times. The improvements in creep strength (measured as a function of temperature) to which the additions gave rise was less pronounced, but nevertheless of practical significance (approximately 20 per cent. in the optimum service range). The most effective additions varied, with the specific alloy, in the range 0·006–0·01 per cent. boron, or 0·05–0·3 per cent. zirconium: higher additions impaired forgeability.

The presence of the additions could be determined microscopically only at the higher contents. Analysis of isolated residues of a grain-boundary phase formed at 1000°C. revealed the phase to be considerably enriched with boron or zirconium.

Experiments with vacuum-melted specimens, and observations on creep behaviour, lead the authors to conclude that boron and zirconium not only have a purifying effect, but also directly influence resistance to deformation.

### Creep-Rupture Properties of Nickel-Chromium Alloys

R. WIDMER and N. J. GRANT: 'The Creep-Rupture Properties of 80 Ni-20 Cr Alloys.'

*Amer. Soc. Mechanical Engineers*, 1959, *Paper* 59-*A*-119; 10 pp.

It is known that the creep behaviour of alloys, as assessed in terms of strength or ductility, can be markedly influenced by minor constituents and the presence of contaminant elements. The investigation now described was undertaken to study the creep-rupture properties of 80–20-type nickel-chromium alloys as a function of temperature, time, alloy preparation (melting in air vis-à-vis *in vacuo*), and small variations in minor constituents and 'tramp' elements.

The composition of the five alloys tested (all of which, after solution-treatment, were held for 24 hours at 1500°F. (815°C.) to ensure precipitation of carbides or nitrides and prevent any ageing effect) are given in the table below.

| % | 1V | 2V | 3A | 4A | 5A |
|---|---|---|---|---|---|
| Ni .. | bal. | bal. | bal. | bal. | bal. |
| Cr .. | 19·17 | 19·59 | 19·45 | 19·98 | 19·96 |
| C .. | 0·02 | 0·03 | 0·02 | 0·02 | 0·02 |
| S .. | 0·011 | 0·003 | 0·003 | 0·002 | 0·004 |
| P .. | ← | ← | <0·003 | → | → |
| Si .. | 0·12 | 1·33 | 1·38 | 1·22 | 0·30 |
| Fe .. | 0·07 | 0·10 | 0·12 | 0·34 | 0·60 |
| Mn .. | 0·11 | 0·27 | 0·01 | <0·01 | <0·01 |
| $H_2S$ group metals | ← | ← | <0·05 | → | → |
| Mn/S ratio | 10 | 90 | 3·3 | <5 | <2·5 |

Alloys 1V and 2V were vacuum-melted; the remainder were air-melted.

Constant-load creep-rupture tests were conducted (mainly in air) at 1200°, 1500° and 1800°F. (650°, 815° and 980°C.). Stresses were selected to produce failure in times ranging from a few seconds to several thousand hours. Elongation and reduction-in-area at fracture were determined, and, in order to assist in the drawing of the log.rupture-time *versus* log.

stress or log.minimum-creep-rate curves, the ruptured specimens were subjected to metallographic examination to determine structural changes. In other experiments the tests were interrupted prior to fracture to permit study of such factors as initiation and progress of intercrystalline cracks and the influence of nitrogen and oxygen contamination from the atmosphere. A close examination of ductility behaviour was undertaken by breaking down the creep curve, as a function of stress and temperature, into primary, secondary, and third-stage creep.

The results indicate that melting variables, minor constituents and contaminant elements play a significant rôle in determining the overall stress-rupture properties of the alloys. The results were, however, not entirely predictable in terms of rupture life or stress for a given rupture time. The vacuum-melted high-silicon alloy was, for example, superior at 1200°F. (650°C.) at high and very low stresses, but not at intermediate stresses. The same effect was observed in the case of measurements of the minimum creep rate, and even the presence of a significant amount of silicon was not found to exert a predictable influence on either the rupture life or the minimum creep rate over a wide temperature or stress range.

The effect of vacuum melting was more pronounced when determined as a function of the ductility behaviour of the alloys in air. While data on first-stage and second-stage creep indicate that the variation between air-melted and vacuum-melted material was small and was not significantly influenced by the addition of silicon, comparison of the alloys, on the basis of total elongation or reduction-in-area at fracture, revealed that ductility values for the vacuum-melted material were two to three times greater than those for the air-melted material, a superiority which was apparently maintained at all three test temperatures and all rupture times.

In comparing the various alloys on the basis of ductility, it is noted that the high-silicon vacuum-melted alloy 2V exhibited significantly higher ductilities. A close examination of the creep curves demonstrated that this difference was due primarily to the amount of elongation occurring during the third stage of creep, and the superior ductility can, it is considered, be related to the elimination of tramp elements normally present in the air-melted alloys. On the other hand, it was found that the ductility of vacuum-melted alloys can be relatively poor if the Mn/S ratio and/or the silicon content is slightly changed.

*See also*

**Influence of Atmosphere on the Creep-Rupture Behaviour of Nickel-Chromium Alloys**

R. WIDMER and N. J. GRANT: 'The Rôle of Atmosphere in the Creep-Rupture Behaviour of 80 Ni-20 Cr Alloys.'

*Amer. Soc. Mechanical Engineers*, 1959, *Paper* 59-*A*-122; 5 pp.

In the test programme described in the paper referred to in the preceding abstract, it was noted that the differences in rupture life, or stress for a given life, were not consistent over the temperature range studied, and that alloys 1V, 2V and 3A displayed higher strengths within narrow regions of temperature and rupture time. Differences in ductility values, which were most pronounced, were attributed to minor constituents and the structure of the grain boundaries, and vacuum melting is therefore deemed of significance in this respect. The test atmosphere, like vacuum melting, was also expected to have a significant effect on creep-rupture behaviour, and in this paper the authors discuss that phase of their investigation covering the influence of atmosphere on the rupture properties of the three alloys.

The specimens studied were subjected to metallographic examination after creep tests in air (at 1500° and 1800°F.: 815° and 980°C.) and in argon (at 1800°F.). Particular attention was paid to the propagation of intercrystalline cracks.

From the data presented it is shown that an air atmosphere has a strengthening effect. In discussing the results the authors raise the following points:

'These studies clearly showed that much more internal oxidation takes place in the low-silicon alloy than in the high-silicon alloy. The mechanism of strengthening in the high-silicon alloy is that of crack oxidation and nitriding, and internal oxidation to a lesser extent. It is not quite clear why nitrides were not found in the structure of alloy 1V.

'From these results it is clear that the intercrystalline cracks which form extensively during the last stage of creep expose metal to oxygen and nitrogen from the atmosphere.

'Oxide and nitride formation on these surfaces, and especially at the radius of each crack, obstruct and delay crack propagation. Simultaneously internal oxidation leads to further strengthening. The action of either or both mechanisms causes relative strengthening and results in the re-establishment of a second period of decreasing creep, followed by a second period of last-stage creep which brings about failure. Of special interest is the apparent interconnexion of the great bulk of the intercrystalline cracks.

'Because of the difference in rate of crack formation and crack propagation, the vacuum-melted alloys (1 and 2V) and the air-melted alloy (3A) show different amounts of cracked boundary area to the atmosphere at any one time during the creep process. This would necessarily stipulate a difference in subsequent strengthening by reaction with the atmosphere.'

**Properties of Nickel-containing Alloys and Steels under Conditions of Rapid Heating and Loading**

J. R. KATTUS and D. W. MCDOWELL: 'Strength of Structural Alloys under Rapid Heating and Loading.'

*Amer. Soc. Testing Materials*, 1960, *Preprint* 71; 11 pp.

Many aeronautical missiles are designed for a service life of only a few seconds or minutes, during which time certain of the structural components are subjected to rapidly applied stresses and elevated temperatures.

**Condition of Alloys Evaluated**

(*See abstract on p. 211*)

| | |
|---|---|
| Type 301 .. .. | 80 per cent. cold-reduced by rolling at room temperature (−75°C.). |
| Type 304 .. .. | 80 per cent. cold-reduced by rolling at −105°F. (−75°C.); stress relieved 800°F. (425°C.), 24 hours. |
| 'PH 15-7 Mo'<br>Condition CH-900<br>Condition RH-950 | Cold-reduced 60 per cent. at 75°F. (25°C.); aged 900°F. (480°C.), 1 hour; air-cooled.<br>1750°F. (955°C.) 10 min.; air-cooled; −100°F. (−75°C.), 8 hour; 950°F. (510°C.), 1 hour; air-cooled. |
| 'René 41' .. .. | Water-quenched from 1950°F. (1065°C.); tempered 16 hours at 1400°F. (760°C.); air-cooled. |
| 'Inconel X' .. .. | 50 per cent. cold-reduced; aged 20 hours at 1100°F. (595°C.); air-cooled. |
| 'Nimonic 90' .. | 30 per cent. cold-reduced; aged 16 hours at 1300°F. (705°C.); air-cooled. |

Since the load-carrying ability of materials under these conditions may differ considerably from that determined in conventional tension and creep tests, an investigation was made of the tensile and creep strength of several stainless steels and nickel alloys under conditions of rapid heating, rapid loading and short exposures to high temperature and stress. The results of this work, which are presented in the paper, will, it is expected, be of use in the design of missiles.

Six materials were evaluated: the nickel-chromium-molybdenum-cobalt-base alloy 'René 41', the nickel-chromium-base alloy 'Inconel X', the nickel-chromium-cobalt-base alloy 'Nimonic 90', the chromium-nickel precipitation-hardenable steel 'P.H. 15-7 Mo', and two 18-8 stainless steels of A.I.S.I. Type 301 and 304. The conditions in which the materials were tested are shown in the table above.

All the test specimens were sheet material oriented in the longitudinal direction. The Type 304 steel cold-reduced 80 per cent. at −105°F. is an experimental material, representing an initial attempt by Southern Research Institute to develop superior strength in austenitic stainless steels by working them at sub-zero temperatures. The specimens were exposed, within 30 seconds, to test temperatures up to 2000°F. (1095°C.). Tensile determinations were made at a strain rate of 0·1 in. per in. per sec.; creep properties were determined at constant-load levels causing rupture in times ranging from 1 minute to 30 minutes.

The load-carrying capacity of all the alloys increased as the time of exposure to load decreased from 30 minutes to 1 second. The high-strength stainless steels were stronger than the nickel alloys at the lower temperatures, but the nickel alloys retained their load-carrying capacity to higher temperatures, and their short-time creep strengths in the range 1400°–1800°F. (760°–980°C.) were roughly equivalent to those of the stainless steels in the range 1000°–1400°F. (538°–760°C.).

At temperatures up to 1200°F. (650°C.), Type 304 stainless steel (cold-rolled at −105°F. to achieve a yield strength exceeding 350,000 p.s.i. at room temperature) exhibited a higher tensile strength than any commercial grade of stainless steel. The strength of cold-worked structures was superior to that of precipitation-hardened structures at low temperatures and, for short rupture times, at moderately elevated temperatures. With increasing temperatures and times under load, the strengths of the cold-worked structures were approached or exceeded by those of the precipitation-hardened structures.

## Influence of Grain-Size on the Properties of 'Nimocast 258'

F. G. HAYNES and D. R. WOOD: 'Some Effects of Grain-Size on the Mechanical Properties of 'Nimocast 258'.' *Metal Treatment*, 1960, vol. 27, June, pp. 219–23.

One of the recently developed nickel-base alloys, 'Nimocast 258'*, retains, in the cast form, more than 70 per cent. of its room-temperature tensile strength at temperatures up to 900°C. Its high resistance to creep at temperatures up to at least 1000°C. suggests its suitability for gas-turbine applications.

Since it has often been suggested that grain-size in the as-cast condition may significantly influence the mechanical properties of cast alloys, the present work was undertaken to evaluate the effects of grain-size on the room-temperature tensile properties and high-temperature stress-rupture and fatigue properties of 'Nimocast 258'. The investigation covered a range of grain-sizes commonly found in investment castings of types used for gas-turbine components, and was carried out on investment-cast test bars of suitably small section thickness.

Test bars were vacuum-cast from four heats of vacuum-melted stock. Controlled casting temperatures were used to produce pre-determined grain-sizes. All the bars cast at 1340°C. showed an average grain-size not greater than 0·5 mm., as measured in the region of the gauge portion on the cast surface. A casting temperature of 1380°–1400°C. gave specimens varying in average grain-size from approximately 1 to 3½ mm. From the latter series of bars, batches of two different grain-sizes were selected: (*a*) 1–2 mm., and (*b*) 2½–3½ mm. Specimens in the as-cast condition were subjected to tensile tests at room temperature, and to stress-rupture and rotating-bend-fatigue tests at 750°, 870° and 980°C.

* Nominal composition: cobalt 20, chromium 10, molybdenum 5, aluminium 4·8, titanium 3·7, boron 0·01, carbon 0·22, silicon 0·25, manganese 0·25, copper 0·25 max., iron 2 max., per cent., remainder nickel.

The extensive data presented demonstrate the feasibility of controlling the grain-size of investment-cast bars by selection of an appropriate pouring temperature. Variation in room- and elevated-temperature strength over the range of grain-sizes studied was found to be small, a finding which provided reassuring evidence that the degree of variation in grain-size occurring in commercial investment castings of moderate section thickness is unlikely appreciably to affect their mechanical properties. When the grain-size was small, strength tended to be higher over extended periods at 750° and 870°C., but the potential advantage inherent in this result may not, it is considered, justify the risk of unsoundness associated with the use of a low pouring temperature in order to obtain fine-grain castings. In this connexion, other factors also require consideration (e.g., the possible effects of grain-size on resistance to thermal fatigue), and alternative means of refining grain-size without risk of unsoundness may also be worthy of study.

### Influence of Temperature on the Properties of Nickel-base Weld Metals

J. HEUSCHKEL: 'Weld Metals in Nickel-base Alloys.' *Welding Journal*, 1960, vol. 39, June, pp. 236S–46S.

Within the nuclear-power industry there has been a tendency (largely in anticipation of improved resistance to stress corrosion) to substitute, or to consider the substitution of, nickel-base alloys for the chromium-nickel austenitic stainless steels. In view of difficulties experienced in the welding of some members of the A.I.S.I. 300 series of stainless steel, it was deemed appropriate to establish the influence of temperature on the properties of sound nickel-base-alloy weld metals, and so to provide a basis for assessing susceptibility to hot cracking during welding and subsequent heat-treatment or service.

The weld metals studied (see compositions below) were deposited either under an argon shield or using a covered electrode. Butt joints were made in basis metal of a similar composition to the electrode. Tensile tests on 0·357-in.-diameter (9-mm.-diameter) unnotched tensile specimens cut from the centre of the welds were made (at a constant strain rate of 750 per cent. per hour) over a temperature which, except for three of the welds, ranged from −320° to +2200°F. (−196° to +1205°C.). One series of these three welds was tested down to −452°F. (−270°C.); the remaining two were not tested below room temperature. The temperature-dependence of the short-time tensile strength and ductility of the as-deposited weld-metal specimens is shown graphically in the paper. Specimens were subjected to microstructural examination and hardness measurements were carried out.

The author's review of the data presented leads him to conclusions which he summarizes as follows:

'1. The transfer efficiencies of the primary alloying elements across the welding arc in the nickel-base alloys are:

| Element | Shielding | |
|---|---|---|
| | Argon | Covered-electrode |
| Fe | 0·98 | >1·0[a] |
| Cr | 0·96 | >0·95[b] |
| Mn | 0·95 | 0·85[b] |
| C | 0·95 | — |
| Ti | 0·82 | 0·92[a] |
| Si | 0·81 | >1·0[a] |
| Al | 0·79 | 0·51[c] |
| Cb | 0·78 | >1·0[a] |
| Ta | 0·65 | >1·0[a] |

[a] Nett for fortified covering.
[b] Nett for unfortified covering.
[c] Independent of covering fortification.

'2. Any desired composition in nickel-base-alloy weld metals can be secured, by either shielding technique, by properly balancing wire and covering compositions.

**Compositions of Nickel-base Weld Metals**
(*See abstract above*)

| Type | Ni % | Cr % | Fe % | Nb % | Ti % | Ta % | Cu % | Al % | C % | Si % | Mn % | S % | O % | N % | Mo % |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| Ni-Ti* | 96·41 | — | 0·13 | — | 2·21 | — | 0·068 | 0·37 | 0·061 | 0·40 | 0·26 | 0·002 | 0·0025 | 0·0062 | — |
| Ni-Ti† | 94·84 | — | 0·28 | — | 3·12 | — | 0·073 | 0·58 | 0·32 | 0·62 | 0·31 | 0·002 | 0·0019 | 0·0021 | — |
| Ni-Cr-Fe-Nb* | 73·85 | 15·7 | 7·04 | 1·41 | 0·26 | 0·17 | 0·035 | 0·12 | 0·031 | 0·16 | 0·20 | 0·007 | 0·0034 | 0·036 | — |
| Ni-Cr-Fe-Nb† | 73·68 | 15·4 | 7·53 | 1·88 | — | 0·14 | 0·094 | 0·09 | 0·031 | 0·24 | 0·65 | 0·002 | 0·0356 | 0·037 | — |
| Ni-Cr-Fe-Nb† | 72·41 | 14·7 | 8·74 | 3·08 | 0·48 | 0·33 | — | 0·24 | 0·073 | 0·40 | 0·45 | — | 0·030 | 0·034 | — |
| Ni-Cr-Fe-Nb-Mo† | 71·44 | 15·5 | 7·93 | 1·98 | 0·25 | 0·16 | — | 0·15 | 0·041 | 0·45 | 1·44 | — | 0·044 | 0·025 | 0·55 |
| Ni-Cr-Fe-Ti* | 73·16 | 15·0 | 6·91 | 0·47 | 2·36 | (0·26) | 0·08 | 0·75 | 0·048 | 0·43 | 0·58 | — | 0·005 | 0·005 | — |
| Ni-Cu* | 64·77 | — | 0·22 | — | 1·91 | — | 31·93 | 0·09 | 0·026 | 0·45 | 0·45 | 0·003 | 0·0046 | 0·0076 | — |
| Ni-Cu† | 66·07 | — | 0·46 | — | 0·50 | — | 29·68 | 1·09 | 0·064 | 0·20 | 1·81 | 0·004 | 0·0022 | 0·0005 | — |

* Argon-shielded-arc. † Covered-electrode.

'3. Ni-Cu-type alloy welds are low in ductility at all temperatures above 1200°F. (650°C.), but are very ductile below 800°F. (425°C.) and down to liquid-helium temperatures.

'4. Ni-Ti-type alloy welds have a low-ductility dip centered at 1800°F. (980°C.).

'5. Ni-Cr-Fe-Nb-type alloy welds have maximum hot ductility, particularly when argon-shielded.

'6. Welds in the Ni-Cr-Fe series become precipitation-hardened and very brittle at 1400°F. (760°C.) when more than 2 per cent. Ti is added to the deposit, regardless of the arc-shielding media.

'7. Ni-Cr-Fe-Ti-type alloy welds are not only hot-brittle, but are crack-sensitive under nominal levels of restraint.

'8. Ni-Cu-, Ni-Ti- and Ni-Cr-Fe-Nb-type alloy welds have excellent ductility and strength at extremely low temperatures. The yield stress tends to rise with lowered temperatures about in line with accepted values for f.c.c. metals.

'9. Strain hardening below 1000°F. (540°C.) is basically a function of the amount of strain, and, for a given amount of strain, it is a minimum in the Ni-Cu alloys and a maximum in the Ni-Ti and Ni-Cr-Fe-Ti alloys.

'10. All welds in these alloys are nonmagnetic except for the Ni-Ti compositions.'

**Fabrication and Properties of Welded 'Inconel' Pipe**

W. L. FLEISCHMANN and R. F. GURNEA: 'Fabrication of a One-Inch-Thick Ten-Inch-Diameter Welded 'Inconel' Pipe.'

*Amer. Soc. Mechanical Engineers*, 1960, *Paper* 60-*MET*-7; 11 pp.

This report outlines the exploratory experiments involved in, and the success attained in, the application of the Midwest Piping Company's hot-forming process to the fabrication of welded piping in the nickel-chromium-iron-base heat-resisting alloy 'Inconel'. The fabricated pipe had to satisfy (1) the mechanical requirements stipulated for 'Inconel' plate by *Specification Sb*-168 of the A.S.M.E. Boiler and Pressure-Vessel Code, and (2) the quality requirements laid down by *A.S.T.M. A*358-56*T* for Electric-Fusion-Welded Austenite Chromium-Nickel Alloy-Steel Pipe for High-Temperature Service. It was necessary therefore to obtain detailed information, both on the effect of successive heating and cooling cycles on the properties of the pipe formed from plate, and on the quality of the longitudinal weld.

This paper, which describes the relevant investigation, is divided into sections covering: (1) the Midwest Company's process for manufacturing the 'Inconel' pipe; (2) the test programme in which an exploratory study was made of the effects of the manufacturing process on the properties of 'Inconel'; (3) a report on the quality of the pipe; and (4) the results of metallographic investigation of the longitudinal pipe weld.

The process for the manufacture of welded pipe involves the following basic operations: cut blank; heat blank; form the blank into a U-shape; heat; partially close; re-heat; close; machine groove; weld; heat the welded pipe; size; anneal; and machine. To obtain data on the effects of these forming operations, a surveillance pipe was fabricated from 'Inconel' plate and, after each forming operation, sufficient material was removed to provide tensile-test specimens (which after testing were also subjected to metallographic examination). The data presented show that the fabricated pipe satisfied the requirements of the relevant specifications.

The particular 'Inconel' plate employed in the investigation was found to be sensitive to the welding process: welding with coated electrodes, using the tungsten-arc process, produced sound welds, but the heat-affected zone of welds made by the inert-gas-shielded metal-arc process exhibited fissures (which are associated with segregate regions in the basis metal).

**Influence of Elevated Temperatures on the Properties of Stainless-Steel-Clad Plate**

W. H. FUNK: 'Stresses in High-Temperature Corrosive Environments.'

*Metal Progress*, 1960, vol. 77, May, pp. 71-5.

Carbon-steel plate clad with austenitic stainless steel is widely used in the petrochemical industry for the fabrication of reaction vessels handling hot corrosive liquids. At high operating temperatures the clad plate will be subjected to complex stresses. The influence of such conditions on the properties of clad plate is examined in the present paper.

Production methods are briefly outlined, and reference is made to the types of 18-8 and carbon steel commonly used as cladding and backing materials. It is then shown that it is possible, using the known characteristics of the austenitic and carbon steels, to calculate the theoretical elongation and internal stresses which may occur in a clad plate when it is raised to a higher temperature. Under most conditions, the stainless-steel cladding will be restrained due to its attachment to the thicker carbon steel. Internal tension stresses will develop in the cladding material as the temperature of the plate is raised, and, since the internal stresses in the clad plate must be in equilibrium, the stress in the backing material caused by the rise in temperature will be compressive. The magnitude relationship will be similar to the ratio of the thickness of cladding to that of the backing steel (i.e., in a plate clad with austenitic stainless steel to a thickness of 20 per cent., the stress in the cladding material will be four times the stress in the backing material).

Data derived from laboratory tests and service experience are presented to illustrate the effects of high temperatures (and of temperature cycling) on the properties of stainless-steel-clad plate. These data are regarded as suggesting 'that austenitic-stainless-clad steels have predictable behaviour characteristics when subjected to heat. Even though

the stainless cladding material and the carbon-steel backing have different basic properties, the nett effect in a composite plate form does not appear to be detrimental for service up to 1000°F. (540°C.). In observing that the stainless cladding material may very well be stressed at yield-point levels when the material is hot (and even when it is in the cold condition), one must recall that this is not necessarily detrimental to the performance of the complete vessel. After all, great plastic deformations can occur only if the material yields throughout its whole thickness.'

### Glass-bonded Refractory Coatings for Nickel-containing Alloys and Steels

T. E. COOK and B. W. KING: 'Glass-bonded Refractory Coatings for Iron- or Nickel-base Alloys.'
*Defense Metals Information Center, Battelle Memorial Inst., Memorandum* 16, Apr. 25, 1959; 17 pp.

In recent years porcelain-enamel heat-resisting coatings, and their suitability for use on various alloys, have been the subjects of considerable research. The coatings can be classified into two general types: (1) those intended primarily to prevent, often under erosive conditions, oxidation or other corrosive attack at temperatures of 1200°F. (650°C.) or above (some of these coatings have been found to confer improved resistance to fatigue and creep); (2) those developed for thermal insulation or to suppress radiation from the surface. This memorandum, compiled from unclassified literature, relates the iron- and nickel-base materials for which such coatings have been developed to the coating compositions and processing methods and conditions recommended. The available literature does not provide a basis for a general evaluation of the quality of the various coatings, and no test data are therefore included in the memorandum. (The firing temperature for each enamel provides an indication of its refractoriness.)

Information is presented on coatings for, *inter alia*, the following materials: iron; low-carbon steels; chromium-nickel stainless steels (A.I.S.I. Types 316, 321, 347, 302, 304, 309, 310); 'Inconel'; 'Inconel X'; 'Nichrome'; 'Nichrome V'; 'Hastelloy B'; 'Hastelloy C'; 'Hastelloy F'; 'Hastelloy N-155'; '17-22AS'; 'S-816'; 'Armco 17-14Cu-Mo'; and '19-9DL'.

### Creep and Rupture Properties of 'Ni-Vee' Bronze

See abstract on p. 195.

### High-Temperature Properties of 'Cufenloy 30'

See abstract on p. 196.

### High-Temperature Fatigue Strength of Nickel-containing Cast Irons

See abstract on p. 202.

### Sharp-Edge-Notch Tensile Strength of Nickel-containing High-Strength-Steel Sheet

G. B. ESPEY, M. H. JONES and W. F. BROWN: 'The Sharp-Edge-Notch Tensile Strength of Several High-Strength-Steel Sheet Alloys.'
*Proc. Amer. Soc. Testing Materials*, 1959, vol. 59, pp. 837–71; disc., pp. 872–84.

The object of the investigation was 'to evaluate rapidly the relative resistance to high stress concentrations of several ferritic and austenitic sheet alloys of interest for use in thin-walled pressure vessels'. The steels selected for study were representative of five types:

Low-Alloy High-Strength Steels: A.I.S.I. 4340, A.M.S. 6434, 'UHS 280', '300M' and 'X200'.
Hot-Work Tool Steels: 'Vascojet 1000'.
Martensitic Stainless Steels: A.I.S.I. Type 410, '12 MoV'.
Precipitation-Hardening Stainless Steels: 'AM-350', '17-7 P.H.', 'P.H. 15-7 Mo'.
Cold-Worked Austenitic Stainless Steels: A.I.S.I. Types 304L and 301.

Particular emphasis was placed on the determination of embrittlement ranges associated with heat-treatment or cold-work. The sharp-edge-notch tension test used to evaluate the room-temperature notch sensitivity of the sheet steels was designed primarily to permit rapid evaluation of a material's tendency to fail at low stresses in the presence of high stress concentrations. Test procedures are described in detail. The test qualitatively simulates the embrittling action of defects, such as cracks or areas of nil ductility, and it is believed to be sufficiently severe to give meaningful results when applied to the evaluation of materials for present lightweight highly-stressed pressure vessels.

The practical significance of the extensive data derived from the investigation is discussed. They reveal the extreme sensitivity of sharp-edge-notch strength to heat-treatments known to be embrittling, and they are deemed to establish definitely that the tensile properties of smooth test bars provide no direct indication of notch sensitivity. Fully notch-ductile behaviour was not obtained in the case of tempered martensites at tensile strengths above 200,000 p.s.i. (89·5 t.s.i.: 140·5 kg./mm.$^2$), and to base a component design primarily on the tensile yield strength may, it is considered, be a very dangerous procedure. At strengths higher than 200,000 p.s.i., notch sensitivity varied considerably with the steel, the newer ultra-high-strength materials exhibiting considerable embrittlement.

### Influence of Cold Rolling and Stress-Relief Treatment on the Sharp-Edge-Notch Tensile Characteristics of Austenitic Steel Sheet

G. B. ESPEY, A. J. REPKO and W. F. BROWN: 'Effect of Cold Rolling and Stress Relief on the Sharp Edge Notch Tensile Characteristics of Austenitic Stainless-Steel Sheet Alloys.'
*Proc. Amer. Soc. Testing Materials*, 1959, vol. 59, pp. 816–32; disc., pp. 833–6.

Casing materials for solid- and liquid-propellant

**Composition of Steels Studied**

(*See abstract on p. 215*)

| A.I.S.I. Type | C % | Mn % | Si % | P % | S % | Cr % | Ni % |
|---|---|---|---|---|---|---|---|
| 301.. .. | 0·10 | 1·24 | 0·53 | 0·033 | 0·020 | 17·16 | 7·28 |
| 304 L .. | 0·017 | 1·38 | 0·65 | 0·016 | 0·019 | 18·18 | 9·44 |

rockets must exhibit yield strengths in excess of 200,000 p.s.i. (89·5 t.s.i.: 140·5 kg./mm.²), in conjunction with high notch-toughness. The relatively high impact properties of the chromium-nickel austenitic stainless steels at low temperatures having indicated the likelihood of their possessing a high notch-toughness in the presence of severe stress concentrations, the investigation now described was undertaken to contribute to the limited data available on the properties of the steels at the high deformations necessary to produce the yield strength required.

The sheet selected for study was fabricated from two types of steel (see table above).

Smooth and sharp-edge-notch tensile characteristics were established at room temperature and −320°F. (−196°C.) for specimens cold-rolled (involving reductions in the range 20–70 per cent.) to a final gauge of 0·063 in. (1·57 mm.). The effects of stress-relief treatment were studied by tests on specimens of Type 301 steel (cold-rolled to 60 per cent. reduction) and Type 304L steel (cold-rolled to 70 per cent. reduction) which had been heat-treated for 24–72 hours at temperatures in the range 400°–1100°F. (205°–595°C.). In other tests corrosion rates in boiling 65 per cent. nitric acid were determined as a function of time and temperature of stress relief.

At room temperature both steels were characterized by relatively high sharp-notch yield-strength ratios when tested in the longitudinal direction; in contrast, the steels cold-worked more than about 40 per cent. exhibited high transverse notch sensitivity. At −320°F. specimens tested in the longitudinal direction retained relatively high notch toughness, while the transverse notch sensitivity, particularly in the case of Type 301 steel, increased.

Stress-relief treatment for 24 hours in the range 400°–700°F. (205°–370°C.) raised the room-temperature tensile and yield strength of smooth specimens of both alloys, a beneficial effect which was less pronounced at −320°F. In general, the effects of stress relief on notch sensitivity were complex and not entirely attributable to known metallurgical factors. No appreciable reduction in notch sensitivity was produced by any of the stress-relief treatments, and stress relief at temperatures higher than 900°F. (480°C.) resulted in a considerable increase in notch sensitivity at −320°F.

Heating in the temperature range 600°–900°F. (315°–480°C.) produced, in both steels, structural changes which adversely affected room-temperature and −320°F. sharp-notch properties, particularly in the transverse direction. Corrosion rates obtained in boiling 65 per cent. nitric-acid corrosion tests on stress-relieved highly-cold-worked steels were generally low (the maximum rate being associated with stress-relief treatments in the region of 900°F.). It would appear that the carbide precipitation responsible for this maximum in the corrosion rate is also related to the deterioration in the notch properties resulting from heating in the temperature range 600°–900°F.

**Influence of Creep on the Short-Time Mechanical Properties of '17-7 P.H.' Sheet**

J. V. GLUCK and J. W. FREEMAN: 'Effect of Prior Creep on Short-Time Mechanical Properties of '17-7 P.H.' Stainless Steel'.

*Wright Air Development Center, Tech. Report* 59–339, Oct. 1959; 82 pp.

Improvements in the design and performance of high-speed aircraft and missiles have been such that the structures are subjected, for short durations, to temperatures and stresses conducive to creep, and a suitable material of construction must, therethere, not only possess the creep strength to withstand such conditions, but must also retain satisfactory short-time mechanical properties. The study now described comprised one of a series of investigations designed to throw light on the effects, on the short-time mechanical properties of typical aircraft structural materials, of prior exposure to creep at elevated temperatures.

In a previous test programme these effects had been studied in relation to the chromium-nickel precipitation-hardenable stainless steel '17-7 P.H.' in the TH-1050 condition (see abstracts of *Parts* I and II of *Wright Air Development Center Tech. Report* 57–150 in *Nickel Bulletin*, 1958, vol. 31, No. 12, pp. 336–7). The present report is concerned with tests on sheet material in the RH-950 condition (which, involving refrigeration treatment, results in tensile and yield strengths of, respectively, 237,000 and 222,000 p.s.i. (106 and 99 t.s.i.: 167 and 156 kg./mm.²)).

Test specimens were exposed, at 600°, 800° or 900°F. (315°, 425° or 480°C.) for 10, 50 or 100 hours, to stresses selected to achieve 0·2, 0·5, 1 or 2 per cent. deformation during the exposure period. Other specimens were subjected to the same temperatures, for the same times, without stress. Subsequent to exposure the effects of the conditions studied were determined by means of the following series of tests: tensile and compressive tests at room- and the exposure-temperature; tension impact tests

at room- and the exposure-temperature; determination of room-temperature hardness.

Ductility at room- and exposure-temperature was found to have decreased significantly as a result of prior creep at 600°F., and short-time exposure at 800°F. Room-temperature ductility was reduced also by exposure, in the absence of stress, at 900°F. and (for longer exposure times) at 800°F., the degree of prior creep at these temperatures having little effect. Short-time tests at 800° and 900°F., subsequent to creep testing at the same temperatures, showed that ductility had been little affected either by temperature or creep. The tension yield strengths at 600°F. and room-temperature were increased, and the compression yield strengths decreased, by prior creep at 600°F. Ultimate tensile strength and hardness were not significantly altered. Tension impact strength appeared to be decreased by exposure to creep (although the effects observed were erratic).

Except that in the RH-950 condition the steel was initially stronger, maintained its strength better after exposure to creep, and exhibited a greater loss in room-temperature ductility following exposure at 800° or 900°F., the effects of creep were generally similar to those observed for the steel in the TH-1050 condition.

The information available on the steel in the RH-950 condition indicates that the observed effects were due mainly to an ageing reaction (precipitation of a nickel-aluminium compound is suggested) which, at 800° and 900°F., occurred under the influence of temperature alone, but at 600°F. was stress-activated.

**Fabrication of '17-7 P.H.' and 'P.H. 15-7 Mo' Stainless Steel by Bend Rolling, Deep Drawing and Spinning**

C. T. OLOFSON: 'Fabrication of '17-7 P.H.' and 'P.H. 15-7 Mo' Stainless Steel by Bend Rolling, Deep Drawing, and Spinning.'

*Defense Metals Information Center, Battelle Memorial Inst., Memorandum* 18, May 15, 1959; 4 pp.

The precipitation-hardenable chromium-nickel heat-resisting steels 'P.H. 15-7 Mo' and '17-7 P.H.' are more difficult to form than the A.I.S.I. Types 301 and 302 chromium-nickel 18-8 stainless steels. In tensile tests the precipitation-hardening steels exhibit, in the annealed condition, an elongation in the region of 30-35 per cent. (*vis-à-vis* 50-55 per cent. in the case of Types 301 and 302). Moreover the precipitation-hardening steels work-harden more rapidly. Forming procedures for the conventional stainless steels are covered in manuals published by the producers, and the authors recommend that those for Type 301 stainless steel should be used as a guide when forming either '17-7 P.H.' or 'P.H. 15-7 Mo'. Certain modifications may, however, be necessary to compensate the differences in properties, and in the notes comprising this memorandum an outline is given of those required when bend rolling, deep drawing and spinning the steels.

**High-Strength Steels: Properties, Heat-Treatment and Forming Practices**

A. M. HALL: 'Some Considerations in the Use of High-Strength Steels.'

*Amer. Soc. Mechanical Engineers*, 1960, *Paper* 60-*MD*-9; 9 pp.

In the past 15 years several types of steel have been developed capable of exhibiting tensile strengths in the range 180,000-300,000 p.s.i. (80·5-134 t.s.i.: 126·5-220 kg./mm.$^2$): low-alloy hardenable steels; chromium hot-work die steels; martensitic stainless steels; semi-austenitic precipitation-hardenable steels; and cold-rolled austenitic stainless steels. At ultra-high-strength levels the materials tend to lose ductility and toughness, and their mechanical properties become increasingly sensitive to small changes in composition and process variables. Close control of composition must therefore be exercised during melting, and unconventional melting methods may be required (e.g., melting *in vacuo* rather than in air). Heat-treating temperatures and times must be maintained within narrow limits and heat-treating atmospheres must be as inert as possible. Great care must be taken in almost all operations, and special procedures may have to be devised for fabricating and processing the material.

Each of the five sections of this review is concerned with a specific class of high-strength steel. Each section is similar in format: an outline is given of the properties typifying the class of steel under discussion, the forms in which the steels are available are mentioned, and applications are briefly surveyed. Recommended heat-treatment, forming and welding practices are then noted, and the problems involved in this respect, and the precautions necessary, are touched upon. Data exemplifying the author's textual comments are included in the review.

**Arc Welding of High-Strength Steels for Aeronautical Applications**

J. T. NIEMANN, R. P. SOPHER and P. J. RIEPPEL: 'Arc Welding of High-Strength Steels for Aircraft and Missile Structures.'

*Defense Metals Information Center, Battelle Memorial Inst., Memorandum* 27, July 31, 1959; 43 pp.

Their good mechanical properties at room temperature and at temperatures up to 1000°F. (540°C.) render the high-strength steels (including hot-work die steels, and martensitic and precipitation-hardening stainless steels) suitable for many aircraft and missile applications. An important consideration in the selection of these steels for a particular application is their ability to be fabricated by welding. The Defense Metals Information Center has accordingly made a survey of relevant information, and the data available on arc welding are collated in the present memorandum. (The search for information is continuing, and it is expected that many more data will be available in the near future. Consequently this memorandum should be considered as containing only preliminary information.)

The data presented relate to the welding characteristics of, and the mechanical properties of welds in, the following steels: 'Crucible 56', 'Crucible 218', 'Vascojet 1000' (hot-work die steels); 'P.H. 15-7 Mo', '17-4 P.H.', '17-7 P.H.', 'AM-350', 'AM-355' (chromium-nickel precipitation-hardening semi-austenitic stainless steels); A.I.S.I. Types 419 and 422, '12MoV' (martensitic stainless steels).

### High-Temperature-Corrosion Problems in the Reduction of Nickel-Copper Ores

R. J. LAW: 'High-Temperature-Corrosion Problems in the Reduction of Nickel Copper Ores.'
*Corrosion*, 1960, vol. 16, June, pp. 286t-8t.

In the Sudbury area of Ontario, The International Nickel Company of Canada, Ltd., mine, mill and concentrate as much as 40,000 tons of nickel-copper ore a day. The Copper Cliff smelter works consists of two main circuits: (1) the nickel circuit (for the smelting of nickel concentrate and the production of a high grade nickel-copper matte), and (2) the copper circuit (for the smelting of copper concentrate and production of blister copper). An auxiliary blast-furnace circuit treats direct ore and revert material. In this paper the author discusses (with emphasis on the materials of construction which have proved most suitable for the various applications) some of the corrosion problems involved in the high-temperature operations at the plant.

Relevant equipment in the roasters, drag-line conveyors, reverberatory furnaces, converters, sinters, and hot crushers is reviewed. Materials to which reference is made include: nickel-chromium martensitic white cast iron; S.G. iron; 28-3, 28-8 and 25-12 chromium-nickel heat-resisting steels; 19-11-3 chromium-nickel-molybdenum steel; chromium stainless steels; and a 46-26-5 nickel-chromium-tungsten alloy.

### Survey of Materials for the Supercritical Water Reactor

M. C. UDY and F. W. BOULGER: 'Survey of Materials for Supercritical Water Reactor.'
*Battelle Memorial Inst., Report BMI*-890, Nov. 27, 1953; 52 pp. (Recently de-classified.)

The survey, the results of which are presented in this report, was initiated to assess the properties of likely materials in relation to their suitability for use in various locations of the supercritical water reactor. The general objectives were: to suggest materials of possible interest; to locate data on materials already under consideration; and to point out materials which should be eliminated from consideration.

Nominal compositions, usual heat-treatments, density and thermal-neutron-absorption cross sections are tabulated, and data on tensile strength, 0·2 per cent. offset yield strength, 1000-hour rupture strength, endurance strength, tensile elongation, Young's modulus, coefficient of thermal expansion, and coefficient of thermal conductivity are presented as a function of temperature.

The materials were evaluated in relation to four applications:

(1) Fuel-element tubes for use in the range 400°-1400°F. (205°-760°C.).

(2) Shell materials for use at 400°-800°F. (205°-425°C.).

(3) Low-temperature (inlet) piping for use in the range 400°-700°F. (205°-370°C.).

(4) High-temperature (outlet) piping for use at 800°-1200°F. (425°-650°C.).

Materials covered include: A.I.S.I. 4340 nickel-chromium-molybdenum low-alloy steel; 'Timken 17-22-A', '17-22-AS', '17-22-AV'; A.I.S.I. Types 310, 347 and 410 stainless steels; 'Crucible 422' stainless steel; 'Jessop H46'; 'R.ex 448'; '17-4 P.H.': '17-7 P.H.': 'Stainless W'; 'A-286'; 'Tinidur'; 'Discaloy'; 'Nimonic 75'; 'Nimonic 80; 'Nimonic 80A'; 'Hastelloy C'; 'Hastelloy X'; 'Inconel X'; 'Cooper Alloy V2B'; various titanium alloys.

### Causes of Failure in Equipment Used in Chemical Plant

G. A. NELSON: 'Criteria for Selecting Metals Used in Chemical Plants.'
*Metal Progress*, 1960, vol. 77, May, pp. 80-8, 134, 164.

A survey of eight chemical plants and nine oil refineries, carried out to determine the extent to which failure of materials of construction contributes to plant shutdown and maintenance, revealed that there are about 60 ways in which metals or equipment can fail under exposure to the rigorous environments of the chemical industry. Most of the failures could be traced to metallurgical phenomena, and classification of the information, according to the principal reason for the failure, showed that two categories were of predominant importance: those covering the effects of temperature and corrosion. The most important factor was temperature, since it, more than any other, produces changes in steel structures which render them unsuitable for use except in a narrow range. The presence of stress or a gaseous environment magnifies the effects of temperature, and these two variables were therefore included in an examination of the effects of temperature on the materials of construction.

The following factors were established as of significance:

*Temperature:* transition temperature (brittle fracture); ageing; 885°F. embrittlement; graphitization; sigma formation.

*Temperature+Stress:* Creep; thermal stresses; thermal fatigue.

*Temperature+Gases:* Air and oxidizing gases (such as steam, carbon-dioxide and furnace gases); hydrogen sulphide; hydrogen; hydrogen sulphide + hydrogen ; nitrogen ; carbon monoxide ; vanadium pentoxide.

In this paper the author reviews the problems and failures associated with each of the factors listed above, and briefly considers the materials or means

by which such failures can be prevented. Materials discussed include carbon and low-alloy steels, chromium-nickel and straight-chromium stainless steels, and nickel-base alloys.

### Use of Nickel-base Alloys in Chemical Plant

W. KATZ: 'Recently Developed Nickel-base Alloys for Use in Chemical Plant.'

*Werkstoffe u. Korrosion*, 1960, vol. 11, June pp. 357–69.

This review of nickel-base alloys used, or proposed for use, in chemical plant is in two sections: in the first section, the author reviews the relevant properties of the commercially available nickel-molybdenum-, nickel-molybdenum-chromium-, and nickel-chromium – iron – molybdenum – base corrosion – resisting alloys; the second, and much briefer, section is concerned with nickel-base alloys which offer a combination of corrosion- and heat-resistance at high temperatures.

In the main part of the paper alloys of nickel-molybdenum, nickel-molybdenum-chromium, and nickel-chromium-iron-molybdenum type are discussed in relation to their corrosion-resistance in various acids, their mechanical properties, workability, and weldability, their metallurgical and structural characteristics, and their corrosion behaviour. (In this last respect, attention is directed to the alloys' electro-chemical behaviour, and to the factors influencing their susceptibility or resistance to intergranular corrosion.)

In the second part of the paper the author draws attention to the advantages offered to the chemical industry by low-carbon nickel and the nickel-base alloys 'Hastelloy N', 'Hastelloy X', 'Inconel' and 'Inconel X'.

Throughout the review the points raised in the text are supplemented and illustrated by numerous graphical and tabular data.

### Corrosion of Heat-Resisting Stainless Steel by Residual-Oil Ash

M. H. LEIPOLD: 'Mechanisms of Corrosion of 310 Stainless Steel by Vanadium Oil Ashes.'

*Thesis, Ohio State University*, 1958; 80 pp.
Available from University Microfilms, Inc., Ann Arbor, Michigan.

In this study of the mechanism involved in the corrosion of materials exposed to the products of combustion in residual-oil-burning power plant, the corrodent, $Na_2O.V_2O_4.5V_2O_5$ (designated $NV_6$), was selected for investigation as being a principal factor contributing to vanadium corrosion, and because it enabled the study to be confined to one corrodent. A.I.S.I. Type 310* stainless steel was selected as the basis material because of its extensive use in high-temperature applications.

* Compositional limits: carbon 0·25 max., silicon 1·5 max., manganese 2 max., phosphorus 0·045 max., sulphur 0·03 max., chromium 24-26, nickel 19-22, per cent.

Specimens were exposed to the corrodent, for various times, in the temperature range 1100°–1700°F. (595°–925°C.). The reaction products were studied by X-ray diffraction and by chemical analysis. Thermogravimetric analysis was employed to obtain data on the effects of the following factors on the kinetics of the reaction: (1) the three major alloying elements of the steel; (2) the composition of the ash; (3) the temperature of the reaction. The influence of the reaction products on subsequent corrosion was also studied.

The experimental procedures are described in detail, and the results, from which the following conclusions are drawn, are presented *in extenso*:

'1. The rate-controlling factors in the corrosion of 310 stainless steel by $NV_6$ progress through three time-dependent changes.

'2. There is an initial diffusion-controlled period during which the formation of an oxide layer is rate-controlling. During this period iron is depleted from the surface of the metal.

'3. Second, a period exists during which the concentration of iron in the surface of the metal becomes rate-controlling, and the rate of corrosion falls below that predicted by diffusion alone.

'4. Finally, an equilibrium concentration gradient in the metal obtains, and the rate again becomes diffusion-controlled with a lower diffusion constant than for the initial period.

'5. The nature of the reaction products which form under these conditions varies according to temperature of formation.

'6. Below 1450°F. (790°C.), the reaction products consist of orthovanadate and corundum structures.

'7. Above 1450°F. (790°C.), the reaction products consist of spinel and corundum structures.

'8. The corrosion rates of the pure constituents of 310 stainless steel (viz., iron, nickel, and chromium) are not affected by the addition of the corrodent. The same diffusion-controlled mechanisms are controlling with or without the corrodent.

'9. The preferential attack on the iron in the corrosion of 310 stainless steels suggests little probability for success in alloying ferrous alloys to obtain a more resistant material.'

### Fire-Side Corrosion in Oil-Fired Boilers

L. K. RENDLE, R. D. WILSDON and G. WHITTINGHAM: 'Fire-Side Corrosion in Oil-Fired Boilers.'

*Combustion*, 1959, vol. 31, Aug., pp. 30–42.

Depending on the temperature of the heat-exchange surfaces affected, two distinct types of corrosion occur in boilers fired by residual fuel oils: low-temperature corrosion of economizers, air heaters and stacks is associated with the condensation of sulphuric acid from flue gases when the temperature of the metal falls below the dewpoint of the acid; high-temperature corrosion is to a large extent governed by the composition and quantity of ash formed during combustion.

In the first part of this paper, the authors describe work concerned with the assessment, using a small-scale rig, of the resistance of a range of superheater and structural steels to corrosion resulting from the combustion of low- and high-vanadium fuel oils. These studies formed part of a comprehensive research programme to determine the influence of fuel type, combustion conditions, and additives on oil-ash corrosion of steels at elevated temperatures. The second section reviews the results of full-scale and pilot-scale tests designed to provide information on the degree of formation of sulphuric acid in oil-fired boilers, and on the efficacy of operational and other methods of controlling low-temperature corrosion.

Data on the effects of dolomite injections on the corrosion behaviour of superheater steel operating at elevated temperatures are reported, and an outline is given of the findings of a study carried out to investigate the feasibility of using additives (including ammonia) as a means of combatting low-temperature corrosion under laboratory conditions.

The following steels and alloys were studied in Part I of the investigation: 18-11 and 17·5-11 Cr-Ni-Nb steels; 13·5 Cr steel; 2¼-1 Cr-Mo steels; 1½ Cr-Mo steels; 20-7, 25-20 and 25-12 Cr-Ni steels; 60-12 and 53-18 Ni-Cr alloys.

### 60-40 Chromium-Nickel Alloy with Improved Resistance to Residual-Oil-Ash Corrosion

'New 60% Chromium-40% Nickel Alloy.'

*INCO Magazine*, 1960, vol. 27, No. 9, pp. 30–1.

In recent years the corrosion problems associated with the combustion products of residual fuel oils used in industrial power plants have been the subject of much research (see, for example, reports of a comprehensive investigation of the resistance of various alloys and steels to such attack abstracted in *Nickel Bulletin*, 1960, vol. 33, No. 1–2, pp. 23–4; No. 4, pp. 86–7). The present article draws attention to a 60–40 chromium-nickel alloy which, developed by the U.S. Navy, has been found to offer a service life four times that of materials previously used to withstand oil-ash corrosion.

Service tests aboard the U.S.S. 'Saratoga' showed that the performance of dogbone-type superheater tube supports in 25-12 chromium-nickel stainless steel was extremely poor, particularly in the hotter regions of the superheater cavities, due to severe corrosion by vanadium-bearing fuel-oil ash. In 1957 four tiers of dogbones fabricated from the 60–40 alloy were installed on the firebox side of several boilers, and examined after periods of up to 7000 hours' steaming. None of the 536 castings installed exhibited significant wastage or evidence of slagging, even in the hottest portions of the superheater cavities, and none required replacement. On the basis of visual observation, it was estimated that the 60–40 supports would be capable of at least 10,000 hours more service (in contrast, the average life of 25-12 steel supports would appear to be in the region of 3000–4000 hours).

In view of these findings the U.S. Bureau of Ships issued, in September 1958, a directive recommending that replacement spares of 60–40 chromium-nickel supports be procured for 36 naval vessels.

*See also*

### Physical and Mechanical Properties of Commercially Cast 60-40 Chromium-Nickel Alloy

D. W. McDOWELL and J. R. MIHALISIN: 'Physical and Mechanical Properties of Commercially Cast 60Cr–40Ni Alloy.'

Issued by INTERNATIONAL NICKEL COMPANY, INC., Aug. 1, 1960; 15 pp.

The first large contract for producing castings in the 60–40 chromium-nickel alloy referred to in the preceding abstract was placed with the National Alloy Division of Blaw-Knox Company. The nominal composition of the alloy (which was melted in a direct-arc melting furnace under an argon atmosphere) was: chromium 58–62, silicon 1·02 max., carbon 0·05 max., manganese 0·3 max., sulphur 0·02 max., phosphorus 0·02 max., per cent., remainder nickel. The total content of elements other than nickel and chromium was not to exceed 1·5 per cent.

In the main section of this paper the authors summarize the properties exhibited by two commercial heats of the alloy, one of low carbon (0·011 per cent.), the other of high carbon (0·14 per cent.) content. The microstructural features of the alloys are discussed, and tabular and graphical data are presented on: response to heat-treatment; coefficient of thermal expansion; tensile properties at ambient and high temperatures; stress-rupture properties; oxidation-resistance; and resistance to corrosive environments typical of those encountered in boilers fired by residual fuel oils.

Universal Cyclops Steel Corporation has produced, by vacuum melting and hot rolling, a wrought modification of the casting alloy (see following composition): chromium 50·07, nickel 47, iron 1·89, aluminium 0·69, titanium 0·57, zirconium 0·38, carbon 0·17, silicon 0·12, phosphorus 0·1, nitrogen 0·042, manganese 0·03, sulphur 0·014, per cent. The second section of the paper contains five tables covering the tensile, impact, and stress-rupture properties of the wrought alloy, its oxidation-resistance, and its hardness after various heat-treatments.

### Corrosion-Resistance of Nickel-containing Materials in a $HF/H_2O$ Atmosphere at High Temperatures

C. F. RITCHIE and E. K. TETER: 'The Corrosion-Resistance of Several Alloys in an $HF/H_2O$ Atmosphere at High Temperatures.'

*U.S. Atomic Energy Commission, Report NYO* 1330, Jan. 22, 1952; 15 pp. (Recently de-classified.)

The test programme reported was carried out with the aim of determining alloys or steels suitable for use as materials of construction in $UF_4$ process equipment.

Specimens of each alloy were placed in a small test box containing $UO_2$, and then exposed, at 1070°F. (575°C.) for 20 hours, to a $HF/H_2O$ atmosphere (the HF concentration was gradually increased from 60 per cent. to 100 per cent.). After 20 hours the test box was removed, and the specimens were air-cooled to room temperature. This procedure was repeated, on an average, three times a week.

The materials selected for investigation, and on which corrosion data are presented, are classified as follows:

*Cobalt-base Alloys:* 'L605', 'Multimet 95', 'N-155', 'S-590'.

*Nickel-Chromium-base Alloys:* 'Inconel' (fluoride-coated); 'Electro-Alloy H3'; 'Illium G' (cast and rolled); 'N-238'; 'Hastelloy C'; 'D-10' (cast); 'Chromel P'; 'Hundred Metal'; 'Nichrome V'; 'Hastelloy C' (stabilized).

*Chromium-Nickel Stainless Steels:* A.I.S.I. Type 310; '16-25-6'.

*Copper-Nickel Alloys:* 'Ambralloy 917'; 'Monel' (fluoride-coated); 'Super Nickel (702)'; '326 Monel'; 'Advance Metal'; 'Copel'.

*Miscellaneous Alloys:* 'Z-Nickel'; 'Stellite No. 6'; malleable cast iron.

*Precious-Metal Alloys:* 95 Pt-5Ir; 95 Pd-5Au on a nickel (2% Mn) base; 95·5 Pd-4·5 Au; 60 Pd-35 Ag-5 Au; 60 Pd-40 Cu; 80 Au-20 Pd.

The test results indicate that, as a group, the nickel-chromium alloys exhibit the highest corrosion-resistance when strength at high temperatures is a prerequisite. Other alloys gave excellent results with respect to corrosion-resistance, but lack of strength at high temperatures, or cost considerations, preclude their use for production equipment.

### Dissolution of Nickel in Acidified Ferric and Ceric Solutions

See abstract on p. 190

### Microscopical Identification of 18-8, 18-8Ti and 18-8Nb Stainless Steels

R. P. WENTWORTH: 'Inclusions Help Identify Stainless Steels.'

*Metal Progress*, 1960, vol. 77, Mar., pp. 125-6.

In this short article the author describes, and discusses the advantages of, a metallographic technique developed, by J. Bishop and Co., Malvern, Pa., as a means of rapidly identifying the three types of stainless steel (A.I.S.I. Types 304, 321 and 327) from which the Company produces tubing.

The technique is based on the fact that stabilized austenitic steels (Types 321 and 347) contain characteristic massive inclusions which are not dissolved during high-temperature annealing at 1900°-2000°F. (1035°-1095°C.). The inclusions present in Type 321 (titanium nitrides) are highly angular (frequently cubic in shape), with dark borders. Under white light, the colour varies from pale orange to pink. Type 347, on the other hand, contains niobium nitride-carbide inclusions which, while somewhat similar to the inclusions in Type 321, have certain features which provide a means of distinguishing the two stabilized types of stainless steel. The niobium-type inclusions, although massive, lack the angularity which is typical of the titanium inclusions. Under white light, moreover, niobium inclusions lack the characteristic colour of the titanium-nitride particles; in fact their colour almost matches that of the surrounding matrix.

A mirror finish is not necessary to delineate the inclusions satisfactorily, and the total time required to polish and identify one specimen is only a minute or so.

### Thermal Expansion of Stainless Steels

B. E. NEIMARK: 'The Thermal Expansion of Stainless Steels.'

*Fiz. Metal. Metalloved.*, 1959, vol. 7, No. 3, pp. 389-94. Translated in *Physics of Metals and Metallography*, vol. 7, No. 3, pp. 64-9.

The work reported was initiated to obtain experimental evidence in support of formulae permitting calculation of the coefficient of linear expansion of a specific type of stainless steel over a given range of temperature.

The compositions of the steel studied are reproduced in the table on p. 222. The data derived from determination of the coefficients of linear expansion of the austenitic and martensitic grades indicate that values for steels of a given type are virtually independent of variations in the composition of the steel. The formulae evolved by the author are shown to permit calculation of the average coefficient of linear expansion with an accuracy of $\pm$ 5 per cent. That covering the coefficient of chromium-nickel steels (chromium 15-22, nickel 8-15, other alloying elements 3 max., per cent.) over the range 100°-900°C. is given below:

$$\bar{\alpha} \times 10^6 = 14 \times 6 + 1 \cdot 59 \times 10^{-2} t - 0 \cdot 23 \times 10^{-4} t^2 - 0 \cdot 013 \times 10^{-6} t^3 \; 1/°C.$$

### Recrystallization of an 18-12-type Austenitic Steel

H. BORCHERS, K. BUNGARDT and D. PUSCH: 'Study of Recrystallization in an Austenitic Chromium-Nickel Steel with Special Reference to the Annealing Time.'

*Archiv f.d. Eisenhüttenwesen*, 1960, vol. 31, Apr., pp. 237-42.

The recrystallization of a vacuum-melted austenitic steel of the following composition was studied as a function of the degree of deformation and the time and temperature of subsequent heat-treatment: carbon 0·02, silicon 0·27, manganese 0·03, phosphorus 0·013, sulphur 0·025, chromium 17·43, nickel 12·4, molybdenum 0·1, nitrogen 0·014, per cent.

**Composition of Steels Studied**

*(See abstract on p. 221)*

| C % | Si % | Mn % | Cr % | Ni % | W % | Mo % | V % | Ti % | Nb % | B % | $N_2$ % | Zr % |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 0·10 | 0·59 | 0·43 | 15·3 | 12·3 | 2·76 | 0·72 | — | — | — | — | — | — |
| 0·10 | 0·46 | 0·45 | 13·9 | 13·8 | 2·15 | 0·52 | — | 0·45 | — | — | — | — |
| 0·07 | 0·46 | 0·57 | 14·95 | 16·45 | 2·63 | 0·8 | — | — | 0·86 | 0·01 | — | — |
| 0·06 | 0·53 | 0·55 | 15·58 | 16·45 | 2·6 | 0·75 | — | — | 0·83 | — | 0·1 | — |
| 0·06 | 0·47 | 1·56 | 18·0 | 13·1 | — | 0·68 | 0·51 | 0·58 | — | 0·01 | — | — |
| 0·17 | — | — | 12·0 | 0·65 | — | 1·0 | 0·3 | — | — | — | — | — |
| 0·17 | — | — | 12·0 | 0·65 | 1·0 | — | 0·3 | — | — | — | — | — |
| 0·17 | — | — | 12·0 | 0·65 | — | — | — | — | — | — | 0·1 | 0·6 |

The influence of the annealing temperature was studied, in increments of 100C.°, over the range 20°–1400°C. Specimens were annealed for 24 hours at temperatures up to 500°C., and for times ranging from 15 seconds (and in some cases 1 second) to 2 hours at higher temperatures. Before deformation the grain-size of the steel was in the region of 50$\mu^2$. Data are presented illustrating the variation in structure, grain-size, hardness, and (in the range 20°–500°C.) magnetic saturation.

Some slight recovery was determined after annealing for 24 hours at 500°C. Recrystallization occurred within seconds at high temperatures, and the data presented indicate how, at deformations above the critical range, a fine grain-size can be attained either by annealing for short times at high temperatures or for longer times at lower temperatures. The annealing times required for the attainment of a given grain-size are recorded (as a function of degree of cold-working and annealing temperature) in a table which is reproduced in the right-hand column. The technical advantages and limitations of this table are discussed by the authors (and it is emphasized that account must be taken, *inter alia*, of the fact that austenitic steels may become sensitized to intergranular corrosion as a result of heating at certain temperatures).

| Temperature | Grain-Size | Annealing time to achieve the relevant grain-sizes after a deformation of: | | | | | | |
|---|---|---|---|---|---|---|---|---|
| °C. | $\mu^2$ | 2% | 5% | 10% | 15% | 30% | 50% | 70% |
| 700 | 6—10 | | | | | | | 2h |
| | 11—20 | | | | | | 2h | |
| | 40—50 | | | | | 2h | | |
| 800 | 6—10 | | | | | | | 1′ |
| | 11—15 | | | | | | | 10′ |
| | 16—30 | | | | | | 10′ | ½h |
| | 31—50 | | | | | 10′ | ½h | 2h |
| | 51—70 | | | | | 2h | | |
| | 100—200 | | | | 2h | | | |
| 900 | 15—30 | | | | | | 30″ | 15′ |
| | 50—60 | | | | | 30″ | | 10′ |
| | 90—110 | | | | | 1′ | 10′ | |
| | 120—150 | | | | 10′ | ½h | ½h | ½h |
| | 151—250 | | | | ½h | 2h | 2h | 2h |
| | 251—500 | 2h | ½h | 10′ | 2h | | 2h | |
| | 500—700 | | 2h | 2h | | | | |
| 1000 | 10—20 | | | | | 15″ | 15″ | 3″ |
| | 40—60 | | | | | | | 15″ |
| | 61—100 | | | | | 30″ | 30″ | 30″ |
| | 101—200 | | | | 15″ | 60″ | 60″ | 60″ |
| | 201—300 | | | | 1′ | | | 10′ |
| | 301—400 | | | 15″ | | 10′ | | 30′ |
| | 401—600 | 10′ | | | ½h | ½h | 10′ | |
| | 601—1000 | ½h | 10′ | 10′ | | | | |
| | 1001—1500 | 2h | | | | | | |
| 1100 | 20—40 | | | | | 3″ | | 3″ |
| | 41—60 | | | | | | | |
| | 100—300 | | 15″ | | 15″ | 1″ | 15″ | 15″ |
| | 301—600 | | 30″ | 60″ | 30″ | 30″ | 30″ | 30″ |
| | 601—1000 | 30″ | | 10′ | | 1′ | | 10′ |
| | 1001—2000 | 10′ | 10′ | ½h | 10′ | 10′ | 10′ | 2h |
| | 2001—3000 | | 2h | | 2h | ½h | | |
| 1200 | 10—20 | | | | | | | 1″ |
| | 21—40 | | | | | 3″ | | 2″ |
| | 100—200 | | | 4″ | | | | 3″ |
| | 201—500 | | | 15″ | 15″ | | | 15″ |
| | 501—1000 | 15″ | 15″ | | | 15″ | 15″ | 15″ |
| | 1001—2000 | 30″ | 30″ | 30″ | 30″ | 30″ | 1′ | 10′ |
| | 2001—4000 | 10′ | 10′ | 10′ | | 10′ | 10′ | |
| | 4001—8000 | ½h | 2h | ½h | 10′ | ½h | | |
| 1300 | 90—100 | | | 2″ | | | | |
| | 101—200 | | | 3″ | | 2″ | | 2″ |
| | 201—300 | | | | | 3″ | | 3″ |
| 1360 | 50 | | | | 1″ | | | |
| | 120 | | | 1″ | | | | |
| 1380 | 70 | | | | 1″ | | | |
| 1400 | 150 | | | 1″ | | | | |

**Recrystallization and Grain Growth in 18-8-Ti Stainless-Steel Superheater Tubing**

F. EBERLE and J. S. MAKRIS: 'Fabrication and Annealing Factors Affecting Grain-Size of 18Cr8Ni-Ti Superheater Materials in Steam Boilers.'

*Amer. Soc. Mechanical Engineers*, 1959, *Paper* 59–*A*–311; 12 pp.

The grain-size characteristics of (SA-213, TP321H) 18–8 chromium-nickel-titanium superheater steels have become of interest to the power-boiler industry in view of the premature swelling in service of fine-grained tubing. The grain-size characteristics of the material have been associated with its creep-rupture strength and performance expectancy, and a grain-size requirement has been incorporated into the

material specification for SA-213 in order to ensure that the tubes are suitably heat-treated. It has, however, been pointed out that such a requirement is not feasible, due to the difficulties involved in evaluating microstructures of irregular mixed grain-sizes and the errors which might occur in grain-size determinations. The experiments now reported were intended to throw light on recrystallization and grain-growth characteristics and the factors influential in this respect.

The compositions of the six heats of steel from which the tubing and bar stock investigated were fabricated all fell within the specification range. A study was made of (1) the influence of plastic deformations (prior to annealing) of 25 and 50 per cent., of tension strains of 1, 3 and 5 per cent. (representing the small deformations involved in tube straightening or superheater fabrication), and of critical strains (present in cold-bent tube strips); (2) the influence of time (5–30 minutes) and temperature (up to 2200°F.: 1205°C.) of annealing; and (3) the influence, on nucleation and grain growth, of the titanium/carbon ratio (6·7, 7·0, 7·29, 7·65, 7·74 and 8·2).

Recrystallization and grain growth were studied by microscopical methods and hardness determinations. Grain-sizes associated with the different conditions studied are illustrated in photomicrographs.

The conclusions drawn from the data presented are quoted below.

'1. The recrystallization process of cold-worked 18Cr8Ni–Ti steel may occur at low or at high temperatures, depending on the magnitude of prior strain or deformation. Large deformations shift the process to lower temperatures. Cold-worked tubing has been found to begin to recrystallize at temperatures as low as 1450°F. (790°C.). On the other hand, annealed material containing strains of 5 per cent. or less may, upon re-annealing, not begin to recrystallize until temperatures as high as 1900°F. (1040°C.) are reached and then cause excessive grain coarsening.

'2. For heavily cold-worked material (25 to 50 per cent. reductions), no correlation has been found between the temperature level of recrystallization and the 2000°–2100°F. (1095°–1150°C.) annealing grain-size. General observations suggested that the most heavily cold-worked material gave a somewhat more uniform grain-size than less heavily worked material.

'3. No correlation could be determined between the temperature level of recrystallization and chemistry variations within specification limits. Under comparative conditions, one material with higher silicon and lower nickel content has recrystallized at a higher temperature and given a smaller 2000°–2100°F. (1095°–1150°C.) annealing grain-size, whereas a second material with relatively high silicon and low nickel content recrystallized at a lower temperature, but also gave a smaller 2000°–2100°F. (1095°–1150°C.) annealing grain-size than the corresponding comparison materials. The Ti/C ratios likewise could not be correlated with the temperature level of recrystallization. However, the material with the lowest Ti/C ratio has consistently displayed a somewhat larger grain-size prior to and after recrystallization, and has also given a larger 2000°–2100°F. annealing grain-size than the materials with relatively high Ti/C ratios.

'4. At the completion of recrystallization of heavily cold-worked material, the grain-size was uniformly small. Upon further heating to higher temperatures, the grain-size increased very slowly and uniformly until a temperature of about 1900°–1950°F. (1040°–1065°C.) was reached. At this approximate temperature level, individual randomly scattered grains began to grow at an accelerated rate, leading to a structure consisting of a range or mixture of grain-sizes ('duplex structure').

'5. The grain-size characteristics of tubing which have been annealed at duplexing temperatures cannot be adequately defined by A.S.T.M. grain-size numbers alone, since the latter give no information on the relative proportions of smaller and larger grains.

'6. Material with high Ti/C ratios has given a predominantly small grain-size at annealing temperatures up to 2000°F. (1095°C.). It seems to be reasonable to expect that some heats with high Ti/C ratios may remain fine-grained at temperatures as high as 2050°F. (1120°C.).

'7. Annealing times between 5 and 15 min. have not produced marked differences in grain-size, but extending the annealing time to 30 min. has caused a noticeable general increase in grain-size.'

### Mechanical Properties of Stainless Steels at Sub-Zero Temperatures

See abstract on p. 204.

### Penetration of Chlorine Ions Through the Surface Film on Austenitic Stainless Steels

H. J. ROCHA: 'The Penetration of Chlorine Ions through the Passivity Films on Austenitic 18/8 Cr–Ni Steels.'

*Werkstoffe u. Korrosion*, 1960, vol. 11, June, pp. 352–6.

It is shown that differentiation between primary and secondary passivity, as suggested by Masing, Heumann and Jesper, can contribute greatly to an understanding of the corrosion behaviour of austenitic stainless steels in neutral and slightly acidic media.

In the potential zone of primary passivity, it is possible to predict whether pitting corrosion is likely to occur. In the zone of secondary passivity, the passivity layers are irreversible and influenced only by surface treatment, and the likelihood of pitting corrosion can be assessed only on an empirical basis. When air is present in chloride-containing water the potential of the steel lies in the boundary region between primary and secondary passivity and is liable to fluctuate above and below the boundary potential.

## Corrosion Behaviour and Passivity of Nickel-Chromium Alloys

A. P. BOND and H. H. UHLIG: 'Corrosion Behaviour and Passivity of Nickel-Chromium and Cobalt-Chromium Alloys.'

*Jnl. Electrochemical Soc.*, 1960, vol. 107, June, pp. 488–93.

Nickel-chromium alloys containing up to 29 per cent. chromium, and cobalt-chromium alloys containing up to 23 per cent. chromium, were vacuum-melted from high-purity constituents, and cast under a helium atmosphere. Specimens were then exposed to the following corrosive media (the cobalt-chromium alloys in the as-homogenized condition, the nickel-chromium specimens after cold-rolling and annealing): de-aerated 1·1 *N* $H_2SO_4$; aerated 1·1, 0·1 and 0·01 *N* $H_2SO_4$; 1·0 *N* $H_2SO_4$ containing 25 g./L. of hydrated ferric sulphate; 1·0 and 0·1 *N* $HNO_3$. Corrosion behaviour was studied as a function of measurements, at 25°C., of corrosion rate, corrosion potential and critical current density for passivity.

In the case of the nickel-chromium alloys *corrosion rates* in de-aerated 1·1 *N* $H_2SO_4$ were low and showed little variation with chromium content. In aerated 1·1, 0·1 and 0·01 *N* $H_2SO_4$, alloys containing more than a critical amount of chromium exhibited a corrosion rate of practically zero, corresponding to the passive condition; higher acid concentrations shifted the passivity limits to higher chromium contents. Corrosion rates in 1·0 and 0·1 *N* $HNO_3$ showed that transition from activity to passivity occurred at chromium contents of, respectively, 7·1–8·9 and 8·9–11·7 per cent. In the case of the 1 *N* $H_2SO_4$ solution containing hydrated ferric sulphate, the initial major critical passive composition was shifted to 4·2–6·6 per cent. chromium.

Variation in chromium content did not result in any systematic variation in the *corrosion potentials* of nickel-chromium alloys in de-aerated 1·1 *N* $H_2SO_4$, but in 0·1 and 1·0 *N* $HNO_3$ the corrosion potential was more or less continuously ennobled with increase in chromium content.

Determination of *critical current densities for passivity* of the nickel-chromium alloys in 0·1 and 1·1 *N* $H_2SO_4$ showed the values to be much smaller for the lower acid concentration, but, in both cases, the curves were of similar shape, and the critical current density decreased sharply as the chromium content increased to the vicinity of 14 per cent.

## Metallurgical Colloquium on Aqueous and Gaseous Corrosion

'Third Metallurgical Colloquium on Corrosion (Aqueous and Gaseous), 1959.'
Published by North Holland Publishing Company, 1960; 239 pp.

This volume contains the proceedings (the text of the 18 papers presented and the discussions to which they gave rise) of the *Third Metallurgical Colloquium on Corrosion* held, at Saclay in 1959, under the auspices of the Centre d'Études Nucléaires de Saclay. The scope of papers of specific or general relevance to nickel-containing materials is briefly indicated in the following abstracts.

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### Development in Theories of Oxidation of Metals

J. BÉNARD: 'Recent Developments in Theories of Oxidation of Metals', pp. 1–7; disc., pp. 7–8.

The review is in three sections. In the first the author comments on the various attempts (Pilling-Bedworth, Cabrera-Mott, Wagner) to evolve a general theory explaining oxidation of metals. In the second it is shown that the fundamental oxidation reactions are frequently masked by secondary phenomena whose rôle often appears decisive in the evolution of the reaction. The paper closes with a discussion of the results of a recent study of some of these phenomena (nucleation, surface diffusion, chemisorption).

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### Diffusion Mechanisms and Gaseous Corrosion

W. J. MOORE: 'Diffusion Mechanisms and Gaseous Corrosion', pp. 19–22; disc., p. 22.

The purpose of the paper is 'to consider, in a general way, some of the important basic mechanisms of diffusion in solids that may be applicable during the processes of gaseous corrosion of metals at high temperatures'.

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### Selection of Alloys Resistant to Oxidation at High Temperatures

J. GENILLOUD: 'Selection of Alloys Resistant to Corrosion at High Temperature', pp. 37–51; disc., p. 42.

The aim of the test programme described was to establish, irrespective of mechanical properties, those of the most promising available alloys which would exhibit adequate oxidation-resistance in air at high temperatures.

Some thirty materials were evaluated, representative of the following types: (1) Iron-chromium alloys; (2) Iron-nickel-chromium-base materials containing (*a*) iron 45, chromium 20–25, nickel 20–30, per cent., or (*b*) nickel 45, iron 17–20, chromium 20–25, molybdenum 8–9, per cent.; (3) Nickel-chromium-base alloys of (*a*) 80–20 type, or of 80–20 type modified by (*b*) titanium or silicon additions, (*c*) aluminium and molybdenum additions, or (*d*) iron and aluminium and/or titanium additions.

Specimens were heated, in an atmosphere of dry air, for 1000–2000 hours at 700°, 800°, 900°, 1000° and 1100°C. Oxidation-resistance was assessed in terms of the decrease in the thickness of the specimens as a function of exposure time.

On the basis of the data presented the author concludes:

(1) That the aluminium-containing nickel-chromium alloys of 80–20 type offer the highest oxidation-resistance at temperatures in excess of 1000°C. Of the alloying additions studied, aluminium appeared to have the most beneficial effect on oxidation-resistance.

(2) For service temperatures in the range 800°–950°C., the choice of alloys with adequate resistance is wider: selection can be made not only from those of 80–20 type, but also from the nickel-rich iron-nickel-chromium group.

(3) Almost all the materials studied are considered satisfactory for service at temperatures below 800°C.

(4) It is emphasized that in the present test programme oxidation-resistance was alone studied. Under operation conditions, such factors as mechanical properties require consideration.

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**Morphology of Oxidation Films**

J. PAÏDASSI: 'The Morphology of Oxidation Films. (I) Precipitation within the Oxides. (II) Micrographical Structure of the Films. (III) Study of Interfaces. (IV) Growth of the Film Induced by the Metallic Substrate. (V) Nucleation and Growth of the Oxides in the Film. (VI) Plastic Deformation in Oxidized Specimens', pp. 71–75; 75–81; 83–9; 89–95; 97–104; 104–10.

In this paper the author demonstrates, on the basis of data obtained during studies of the oxidation behaviour, at high temperatures and atmospheric pressure, of polyvalent metals (iron, copper, magnesium, nickel, uranium) and their oxides, that the usual representation of the oxide film (as a superposition of different oxide layers, micrographically homogenous, consisting of equiaxe crystals, free from mechanical strains, and separated by planar interfaces) should be considerably modified to bring it more into line with reality.

In (I) and (II) the author discusses the micrographical structure of the oxidation film, and considers examples of precipitation in the oxides during cooling from the temperature of formation. Parts (III) and (IV) outline the findings of those stages of the test programme covering study of (1) the morphology of the different interfaces in the oxidation film, and (2) the features of oxide growth induced by the metallic substrate. In (V) particulars are given of the occurrence of an oxide in the form of isolated crystals, and in (VI) the effects of plastic deformation in oxidized specimens are discussed.

The paper contains numerous photomicrographs illustrating the author's findings.

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**Corrosion of Stainless Steels in Aqueous Solutions**

L. COLOMBIER: 'Some Features of Aqueous Corrosion of Stainless Steels', pp. 111–23; disc., pp. 123–5.

In this paper (in which reference is made to a *bibliography* of forty-three items) the author reviews the information available on intergranular, pitting and stress corrosion of 18–8-type austenitic steels. The characteristic features of each type of corrosion are discussed, the theories advanced in explanation of the corrosion mechanisms are commented upon, the rôle of corrosion accelerators and inhibitors is considered, and reference is made to measures recommended as a means of reducing the incidence of these types of attack.

**Relationship between Carbide Precipitation and Susceptibility to Intergranular Corrosion in Stainless Steel**

H. HATWELL: 'Study of the Precipitation of Chromium Carbide responsible for Intergranular Corrosion in Austenitic Stainless Steels', pp. 137–44; disc., pp. 145–6.

The investigation reported originated in the assumption that the susceptibility of an austenitic stainless steel to intergranular corrosion would vary with the form in which the chromium-carbide precipitate $Cr_{23}C_6$ were present. An attempt was therefore made to relate the intergranular-corrosion behaviour of chromium-nickel-molybdenum austenitic steel* to the morphology of the $Cr_{23}C_6$ precipitate and its conditions of growth.

Specimens of the steel were treated (full details of the treatment are given) to produce carbide precipitate of the following types: dendritic, of geometrical (triangular) form, and pulverulent (very fine and of no characteristic form). Each specimen was then subjected to the 65 per cent. boiling nitric-acid test to determine its resistance to intergranular attack.

The results show that precipitation of dendritic carbide at the grain-boundaries renders the steel extremely susceptible to intergranular corrosion, and that this susceptibility is attributable to chromium impoverishment of the interdendritic regions. (Bain's theory of a chromium-depleted zone should, it is considered, be modified in the light of these findings.) It is suggested that the steel is rendered immune to intergranular corrosion when the growth of carbide of dendritic form is inhibited by subjecting the steel to plastic deformation before heat-treatment.

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**Corrosion of Stainless Steels in High-Temperature Water**

E. POUILLARD: 'Corrosion of Stainless Steels by Pure Water at High Temperatures', pp. 147–59; disc., p. 160.

The paper summarizes the results of work carried out (by the Department of Metallurgy and Applied Chemistry of the French Atomic Energy Commission) to determine the corrosion behaviour of ten chromium-nickel stainless steels in high-temperature water, and, hence, to evaluate their suitability for use as materials of construction in water-cooled reactors.

Nine of the ten steels (which were supplied by three different French steel makers) were of 18–10 or 18–10–Mo type, and were either of low-carbon content or stabilized with titanium or niobium. The tenth steel was of low-carbon 20–25 type.

The investigation is described in three sections. In the first, details are given of the test conditions, and of the techniques and apparatus (including two types of micro-autoclave machined entirely from the steel under investigation) used to study corrosion behaviour in high-purity water at 350°C. The second and third sections cover studies of resistance to, respectively, surface corrosion and stress corrosion.

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* Composition: carbon 0·08, silicon 1·1, manganese 2, phosphorus 0·04, sulphur 0·04, chromium 16·5, nickel 13·8, molybdenum 2, per cent.

The surface-corrosion tests, in which specimens were exposed for up to one year in the vapour or liquid phase, indicated that the resistance of the steels to intergranular corrosion was good. Pinholing was the main type of corrosion observed, and the number and depth of the pinholes was found to be related to the inclusions present at the surface.

No signs of stress corrosion were determined, even at the end of one year's exposure, but some of the steels suffered appreciable intergranular attack as a consequence of overpickling.

The author's overall conclusion is that the steels studied exhibit very good resistance to high-purity water at 350°C.

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**Corrosion of 'Inconel' in High-Temperature Water**

H. CORIOU, R. GRALL, M. LE GALL and S. VETTIER: 'Stress-Corrosion Cracking of 'Inconel' in High-Temperature Water', pp. 161–8; disc., pp. 168–9.

In the light of the reported resistance of 'Inconel' to stress-corrosion cracking in high-temperature water (see, for example, paper abstracted in *Nickel Bulletin*, 1960, vol. 33, No. 6, pp. 152–3), the present investigation was carried out to determine the alloy's behaviour when exposed, under stress, to water at 350°C. (and, hence, to evaluate its suitability for use under such conditions in water-cooled reactors).

Specimens of the following four heats of alloy (A, C and D of French, B of English, origin) were studied:

| Heat | C % | Si % | Mn % | Ni % | Cr % | Ti % | Fe % |
|---|---|---|---|---|---|---|---|
| A | 0·02 | 0·28 | 0·66 | 77·5 | 14·3 | — | |
| B | 0·04 | 0·21 | 0·17 | bal. | 15·3 | 0·29 | 7 |
| C | 0·05 | 0·33 | 0·57 | 77·4 | 13·6 | — | 7 |
| D | 0·09 | 0·92 | 0·52 | 79·4 | 19·0 | 0·18 | 0·35 |

Specimens 1·5–3 mm. thick were tested in the following conditions: quenched from 1100°C. and polished with emery paper; annealed at 970°C.; as-cold-rolled. The specimens were subjected to a stress slightly greater than their elastic limit, and then exposed in water at 350°C. for 120 (heat A) or 90 (heats B, C and D) days.

The results presented, which are supplemented by numerous photomicrographs illustrating the condition of the specimens after exposure, are discussed in detail. On the basis of the data obtained, the alloys (whether with or without titanium additions) are reported to be susceptible to serious intergranular attack (involving even complete failure within three months' exposure).

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**'Knife-Edge' Corrosion of Stainless-Steel Weldments**

J. HOCHMANN: 'Knife-Edge' Corrosion of Certain Stainless-Steel Weldments', pp. 171–3; disc., p. 174.

'Knife-edge' corrosion occurs in the heat-affected zones of welds in certain titanium- or niobium-stabilized austenitic steels when exposed to boiling nitric acid. It is characterized, according to the present author, by the following features:

It affects zones which are heated to temperatures in the region of 1300°C. during welding, and occurs in titanium- or niobium-stabilized 18–10 stainless steels, but not in unstabilized stainless steels with the same carbon content.

It occurs not only in untreated welded fabrications, but also in those which have been stabilized or stress-relieved by heat-treatment in the range 800°–900°C.

Susceptibility to 'knife-edge' corrosion is not influenced by sensitivity to intergranular corrosion induced by heating at 500°–800°C. It affects only steels containing more than 0·06 per cent. carbon, and occurs irrespective of variation in the titanium/carbon or niobium/carbon ratios.

In this paper the author discusses the factors which contribute to 'knife-edge' corrosion and attempts an explanation of the mechanism underlying susceptibility to this type of attack.

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**Use of the Potentiostat to Study the Behaviour of Metals in Liquid Media**

Ph. BERGE and P. A. JACQUET: 'Some Examples of the Use of the Potentiostat for Study of the Behaviour of Metals in Liquid Media', pp. 175–82; disc., pp. 182–4.

After a brief description of a modified potentiostat, fitted with an automatic recording device and a movable cell, the authors exemplify its use by discussion of work reported in the literature on intergranular corrosion of stainless steels, and by presentation of the results of work on the corrosion of aluminium-magnesium alloys.

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**Precipitates, Brittleness and Intergranular Corrosion in 18-8 Stainless Steel**

J. PLATEAU, G. HENRY and C. CRUSSARD: 'Precipitates, Brittleness and Intergranular Corrosion in 18-8 Stainless Steel', pp. 185–99; disc., p. 199.

Precipitation of chromium carbide in 18-8-type chromium-nickel stainless steels is associated not only with sensitization to intergranular corrosion, but also with intergranular brittleness at low temperatures. In the investigation reported, the authors have, by means of electron microscopy and microfractography, (1) studied the morphology and origin (inter- or intragranular) of the precipitates formed during annealing of 18-8-type stainless steels, (2) investigated the mechanism of intergranular brittleness, and (3) attempted to establish a relationship between intergranular brittleness and susceptibility to intergranular corrosion.

The influence of carbon content and the annealing time and temperature was studied, and tensile specimens, in different conditions of heat-treatment, were broken at −196°C. and the rupture surfaces then examined by microfractography.

It is shown that intergranular precipitation results in intergranular brittleness. Minimum elongation

values at −196°C. were found to be associated with a prior annealing treatment of about 24 hours at 750°C.: the increase in ductility observed for annealing times longer than 24 hours might, it is considered, be attributable to diffusion of chromium or formation of intracrystalline precipitates. The mechanism of intergranular rupture is, it is concluded, similar to that of ductile rupture, the rôle played by the precipitates sufficing to account for the observed phenomena.

Intergranular-corrosion tests on specimens annealed at 750°C. showed that the annealing time for minimum ductility approximated to that for maximum susceptibility to intergranular attack.

### Corrosion of Stainless Steel by Dilute Nitric Acid at High Temperatures and Pressures

M. L. PEARCE and V. S. GRIFFITHS: 'Stainless-Steel Corrosion by Dilute Nitric Acid at Elevated Temperatures and Pressures.'

*Corrosion Technology*, 1960, vol. 7, May, pp. 142–5, 162.

The resistance of stainless steels to mineral acids at high concentrations has been the subject of much research. Industrial developments (particularly in connexion with the design of homogeneous aqueous nuclear reactors) have now focused attention on the sparsity of information available on the resistance of materials to relatively concentrated solutions of heavy-metal salts with the dilute acid content necessary to prevent their hydrolysis. Such solutions frequently have to be handled at temperatures greatly in excess of their boiling points, and in the presence of corresponding steam pressures. The autoclave tests described by the authors were carried out to obtain data on the behaviour of 18–8 chromium-nickel stainless steel under such conditions.

A study was made of: (1) the resistance of the steel to nitric acid at concentrations ranging from 0·01 to 1·4 per cent. and at temperatures of 300°–350°C.; (2) the effects of introducing, into the 1·4 per cent. acid, 100 p.p.m. of chromium sulphate (or a similar amount of cerium impurity); (3) the resistance of the steel to a solution of 10 g./L. of uranyl nitrate in 0·01 per cent. nitric acid; (4) corrosion in the vapour phase above 1·4 and 0·074 per cent. acids.

In the majority of the tests corrosion was assessed in terms of loss-in-weight. Corrosion rates and weight-loss for passivity in the various concentrations of pure acid are reproduced below.

| Acid concentration | Weight-loss per unit area before passivity (mg./cm.²) | Rate of corrosion after passivity (mg./cm.²/yr.) |
|---|---|---|
| 1·41 | 0·95 | 1·0 |
| 0·366 | 0·37 | 0·9 |
| 0·074 | 0·27 | 1·6 |
| 0·010 | 0·24 | 1·7 |

It is concluded that the corrosion rate for passive conditions is constant for all the concentrations studied (i.e., $1 \cdot 5 \pm 1 \cdot 0$ mg./cm.²/yr.). The time needed for the development of passivity appeared to be dependent on the acid concentration and was longer for the more dilute acid. Despite the fact that passive conditions were reached more quickly in the case of the more concentrated acid, the rate of corrosion was greater in the initial stages, and this led to a greater weight-loss for passivity. The initial rate of corrosion was increased, and passive conditions were reached more rapidly, with increase in temperature, whereas the rate of corrosion after passive conditions had been reached was unaffected by temperature.

The effects of the presence of chromic or ceric ions were similar : the initial corrosion rate was considerably increased and the level of corrosion corresponding to passivity was higher. The rate of corrosion corresponding to passive conditions, however, did not appear to be affected to any extent detectable by the method used.

The corrosion rate after passivity and the weight-loss at the passivity level were both increased by the uranyl-nitrate additions.

Corrosion of stainless-steel specimens exposed to the vapour phase was insignificant; attack at the vapour/liquid interface was severe.

The paper includes details of an electrochemical method of evaluating corrosion, which, involving determination of the variation of the capacitance of a stainless-steel cell with corrosion time, has given encouraging results. The capacitance cell was constructed so that it could be dismantled and reassembled without appreciably changing its cell constant. The stainless-steel electrodes could therefore be detached from a nylon cell spacer unit, corroded in the acid at 300°C., and then reassembled in the cell.

### Influence of Water Cooling on the Corrosion of 18-8-Mo Stainless Steel Exposed to Hot Concentrated Sulphuric Acid

H. HOLDSCHICK and K. BITTNER: 'Laboratory Experiments on the Corrosion-Inhibiting Effect of Cooling Stainless Steels in Hot Concentrated Sulphuric Acid.'

*Werkstoffe u. Korrosion*, 1960, vol. 11, Apr., pp. 216–19.

The experiments reported were initiated to study the feasibility of decreasing the corrosion of 18–8–2 nickel-chromium-molybdenum stainless steel in hot concentrated sulphuric acid by cooling the steel with water.

Two U-bent tube specimens were immersed for 72 hours in the hot concentrated acid. Cooling water was passed through one of the tubes; the other was uncooled and served as a control. In the three phases of the investigation described, the influence of the following factors was determined: (1) the wall thickness of the tube; (2) the temperature (80°–90°C.) and concentration (80-95 per cent.) of the acid; (3) the temperature gradient between the cooling water and the acid.

In view of the considerably lower corrosion rates exhibited by the water-cooled specimens, the results of the investigation are deemed of some practical significance. Cooling would, for example, be economically justifiable if the presence of excessively high temperatures were the only factor preventing the use of an otherwise ideal material of construction.

The effect of the cooling water is attributed to the decrease in temperature at the surface of the tube exposed to the acid: the corrosion rates exhibited by the cooled tubes are in agreement with rates determined in long-time exposure tests at the temperature (45°–60°C.) to which calculations indicate the surface was cooled.

### Influence of Nitrogen on the Properties of 18-8 Austenitic Steel

R. R. BRADY: 'A.I.S.I. Type 304L Stainless Steel with Improved Strength.'

*Proc. Amer. Soc. Testing Materials*, 1959, vol. 59, pp. 774–85; disc., p. 785.

A.I.S.I. Type 304L stainless steel (0·03 per cent. carbon max.), which is the low-carbon counterpart of A.I.S.I. Type 304 stainless steel (0·08 per cent. carbon max.), was developed for use in applications requiring improved resistance to intergranular corrosion. Because of its low carbon content, however, Type 304L steel has lower room- and elevated-temperature strength than Type 304, and the permissible working stresses assigned by the American Society of Mechanical Engineers are lower than for Type 304. The Applied Research Laboratory of the U.S. Steel Corporation has therefore investigated the feasibility of increasing (without impairing intergranular corrosion-resistance) the room- and elevated-temperature strength of Type 304L steel by addition of nitrogen. The results of this investigation are summarized in the present paper.

The specimens tested were machined from 3-in.-thick (7·5-cm.-thick) hot-rolled plate reduced from commercial electric-furnace heats, two of high-nitrogen Type 304L steel (0·1 and 0·13 per cent. nitrogen) and one of standard Type 304 steel (0·04 per cent. nitrogen). Specimens from each sheet of steel were subjected (in the annealed condition or after a sensitizing heat-treatment) to the Huey intergranular-corrosion test. Annealed specimens were subjected to tensile tests at room temperature and various temperatures in the range 200°–1200°F. (95°–650°C.), and creep-rupture tests were conducted at 900°, 1100°, 1300° and 1500°F. (480°, 595°, 705° and 815°C.). Specimens in the annealed condition, or after long-time creep-rupture testing, were submitted to metallographic examination and to Vickers hardness tests.

The conclusions drawn from the data presented are quoted below:

'1. In the annealed and in the annealed-and-sensitized condition, Type 304L stainless steel with about 0·10 per cent. nitrogen has intergranular-corrosion-resistance equivalent to that of normal-nitrogen Type 304L steel (0·04 per cent. nitrogen), as measured by the Huey test.

'2. Except for low tensile ductility at 1200°F. (650°C.), the tensile properties of this high-nitrogen Type 304L steel are about the same, in the range from room temperature to 1200°F., as those of regular Type 304 steel (about 0·07 per cent. carbon and 0·04 per cent. nitrogen).

'3. The creep-rupture strengths of the high-nitrogen Type 304L steels are superior to those reported in the literature for regular Type 304 steel in the range 900°–1500°F. (480°–815°C.). With the exception of the 10,000-hour creep-rupture strength at 900°F., and the 1000- and 10,000-hour creep-rupture strengths at 1500°F., the creep-rupture strengths of the high-nitrogen Type 304L steels are as good as, or slightly superior to, those of the regular Type 304 steel studied in the present investigation. However, the regular Type 304 steel studied has much higher strength, particularly at 900° and 1500°F., than that normally exhibited by Type 304 steel.

'4. In the annealed condition, less delta ferrite is present in the high-nitrogen Type 304L steels than in the regular Type 304 steel, and the microstructural stability of the high-nitrogen Type 304L steels is better than that of the regular Type 304 steel.'

### Influence of Boron on the Susceptibility of Austenitic Steel to Intergranular Corrosion

V. V. LEVITIN and V. I. SYREISCHIKOVA: 'On the Effect of Boron on the Nature of the Carbide Precipitation in Austenitic Steel on Tempering.'

*Fiz. Metal. Metalloved.*, 1959, vol. 7, No. 2, pp. 308-10.

Translated in *Physics of Metals and Metallography*, vol. 7, No. 2, pp. 152–4.

To determine the influence of boron additions on carbide precipitation in an austenitic steel during exposure to temperatures in the sensitization range (and hence to determine the influence of these additions on the susceptibility of such steels to intergranular corrosion), nitrogen-containing chromium-manganese-nickel austenitic steels were annealed at temperatures in the range 950°–1250°C., quenched, and subjected to a sensitization heat-treatment of two hours at 650°C. Susceptibility to intergranular corrosion was assessed in terms of corrosion testing, loss of metallic timbre, increase in electrical resistivity and metallographic examination.

The investigation revealed that the presence of boron retards formation of a carbide network at the austenite grain boundaries. In boron-free steel the two-hour heat-treatment at 650°C. resulted in the formation of a continuous carbide network along the grain boundaries, while in steel containing 0·004 per cent. boron the carbide phase precipitated in the form of separate particles, and the grain boundaries remained clear for a considerable distance. At concentrations below 0·03 per cent. or above 0·01 per cent., however, boron was found to have no effect on carbide formation.

The breakdown of the solid solution at the grain boundaries was retarded, under the influence of

boron, when, prior to quenching, the steel was heat-treated in the range 1075°–1150°C. Use of higher or lower temperatures resulted in the formation of a carbide network, just as in a steel free from boron.

The effect of boron became more pronounced as the carbon content of the steel was lowered: in steel containing 0·06 per cent. carbon, microscopical examination showed that boron had almost completely eliminated carbide formation; at a carbon content of 0·14 per cent., boron had only slight effect.

The effects observed are thought to be explicable in terms of the theory that enrichment of the grain boundaries with boron leads to a reduction of crystal-lattice distortion, and, hence, to an increase in the energy of formation for critical precipitate nuclei.

### Intergranular Corrosion of Nickel-Molybdenum and Nickel-Molybdenum-Chromium Alloys

H. GRÄFEN and G. BÖHM: 'Intercrystalline Corrosion of Nickel-Molybdenum-Chromium Alloys.'

*Zeitsch. f. Metallkunde*, 1960, vol. 51, May, pp. 245–52.

Corrosion-resisting alloys of nickel-molybdenum-chromium-base are widely employed in the chemical industry for applications involving operational conditions too severe for the more conventional chromium-nickel austenitic stainless steels. Service experience has, however, shown these alloys to be susceptible, in certain microstructural conditions, to intergranular corrosion. The investigation reported by the author was initiated in an attempt to throw light on the causes and mechanism of this susceptibility.

To this end, specimens of three alloys (see table below) were solution-treated to obtain a homogeneous structure, and then exposed, for times varying from a few minutes to 4 hours, to temperatures in the range 500°–1350°C. After exposure the specimens were subjected to metallographic examination, and the precipitates present were indentified by X-ray diffraction. Susceptibility to intergranular corrosion was assessed on the basis of the results of an accelerated-corrosion test developed by the authors (the strip or rod specimen is subjected to a bend test after 24 hours' treatment with 10 per cent. hydrochloric acid, susceptibility being revealed by cracking at the grain boundaries).

The corrosion data for each alloy are presented in the form of diagrams indicating, as a function of temperature and time at temperature, weight-loss and susceptibility to intergranular corrosion.

X-ray examination demonstrated that sensitization is induced by precipitation of certain phases: in the case of nickel-molybdenum alloys, nickel-molybdenum compounds are the cause at temperatures below 900°C.; at very high temperatures (as, for example, obtain during welding) a second sensitization zone occurs, this time due to precipitation of molybdenum-rich carbides at the grain boundaries. In the nickel-molybdenum-chromium alloys susceptibility is induced by formation of an intermetallic σ-phase at the grain boundaries.

On the basis of the results obtained, the mechanism of intergranular corrosion is interpreted in terms of molybdenum (or molybdenum and chromium) impoverishment at the grain boundaries. The electrochemical processes during intergranular corrosion are discussed in relation to current-density/potential curves obtained in 15 per cent. hydrochloric acid and in 1*N* sulphuric acid at 65°C.

In considering preventive or remedial measures, the authors suggest that sensitization of the nickel-molybdenum alloys (which occurs in the range 550°–900°C.) can be avoided by ensuring maintenance of the solution-treated condition and by rapid cooling when welding (compositional modifications are also suggested as a means of increasing resistance during welding). Welds in the nickel-molybdenum-chromium alloys must be solution-treated in the range 1200°–1240°C. and quenched if sensitization is to be eliminated.

### Influence of Alloying Additions on Hot Cracking of Chromium-Nickel Austenitic Stainless Steels

F. C. HULL: 'Effects of Alloying Additions on Hot Cracking of Austenitic Chromium-Nickel Stainless Steels.'

*Amer. Soc. Testing Materials*, 1960, *Preprint* 78; 20 pp.

In the investigation reported the author was concerned exclusively with the susceptibility of chromium-nickel austenitic stainless steels to cracking during solidification (e.g., hot cracking of as-deposited weld metal and hot tearing of castings). Although the literature contains numerous qualitative references to the beneficial or detrimental effects of various alloying additions on weld-bead cracking, few authors have assessed tendency to cracking quantitatively, and even in cases where this has been done, the results obtained by one method of testing are often difficult to correlate with those from another. The quantitative effects of different alloying elements on susceptibility to hot cracking can therefore be established only by testing a large number of steels under comparable conditions. Such a comprehensive study is described in the present paper.

| Ni % | Mo % | Cr % | Fe % | W % | Si % | Mn % | V % | Co % | S % | C % |
|---|---|---|---|---|---|---|---|---|---|---|
| 67·0 | 26·9 | 0·2 | 4·9 | — | 0·31 | 0·26 | 0·21 | 0·18 | 0·07 | 0·02 |
| 56·0 | 15·8 | 15·0 | 5·4 | 3·8 | 0·44 | 0·36 | 0·23 | 2·0 | 0·01 | 0·04 |
| 63·4 | 16·1 | 15·4 | 5·2 | — | 0·25 | 0·89 | — | — | 0·005 | 0·04 |

The basis composition studied (a low-carbon, low-nitrogen 16-20 chromium-nickel steel which, even with substantial alloying additions, is fully austenitic in the as-cast, cold-worked or hot-worked conditions) was selected as a means of excluding interference from structural variables. The influence of the following types of alloying element was determined: the basis elements chromium and nickel; the interstitial elements carbon and nitrogen; the deoxidizers manganese, silicon and aluminium; the solid-solution hardeners copper, vanadium, molybdenum and tungsten; the carbide stabilizers titanium, niobium and tantalum; and three other strong carbide- and nitride-forming elements, boron, zirconium and hafnium. The effects of these elements were established quantitatively in terms of the results of the Cast Pin Tear Test (a rapid and inexpensive test which produces cracks in chill-cast pins as they freeze and cool under conditions intended to simulate the restraint imposed during welding of heavy sections). The influence of air- and vacuum-melting was also taken into account.

Full details are given of the steel compositions studied, the test procedures used and the data obtained.

Since the results, presented in the form of a series of curves, were found to agree with qualitative reports of cracking published in the literature, it is considered likely that the Cast Pin Tear Test is capable of predicting the quantitative effects of alloying additions on the hot cracking of deposited weld metal. Summarizing (on the basis of equal weight percentages) the relative beneficial and adverse effects of the seventeen elements studied, the author lists them (starting with the most beneficial and ending with the most detrimental) as follows: nitrogen, molybdenum, manganese, tungsten, chromium, cobalt, vanadium, aluminium, nickel, tantalum, copper, silicon, niobium, titanium, carbon, hafnium, zirconium, and boron.

The data presented are regarded as a suitable basis for the development of fully-austenitic stainless-steel electrodes resistant to hot cracking. In this connexion, the author comments: 'In the base composition, chromium should be as high as possible without giving rise to delta ferrite and sigma; nickel should be as low as possible without introducing delta ferrite. Of the deoxidizers, manganese should be much higher than normally employed in chromium-nickel steels, aluminium is permissible in small amounts, and silicon should be low. Higher strength can be obtained by the addition of the solid-solution strengtheners vanadium, tungsten and molybdenum, the latter two actually increasing resistance to cracking as well as improving strength. Carbon should be low, but nitrogen may be relatively high. If carbide stabilization is required, or even greater strength desired, the preferred addition is tantalum, since both titanium and niobium drastically increase the susceptibility of steels to hot cracking.

'The microstructure, as well as the composition, is an important variable in controlling hot cracking. When there is no objection to ferrite, the maximum possible resistance to hot cracking can be obtained by adjusting chromium and nickel (or the other ferrite- or austenite-forming elements in the alloy) to yield 7 to 10 per cent. delta ferrite in the as-cast structure. If elements are added that lead to the occurrence of a small amount of a second phase (other than ferrite) at the dendrite boundaries, the grain-boundary mobility is restricted, with a resulting increase in susceptibility to hot cracking.'

**Properties and Applications of 'S-G Ni-Resist' Austenitic Cast Irons**

See abstract on p. 202.

## ANALYSIS

**Determination of Nickel in Soil and Laterites**

See abstract on p. 190.

**Photometric Determination of Cobalt in Nickel**

See abstract on p. 191.

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